

Studies on Aluminum–Fly-Ash Composite Produced by Impeller Mixing

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE OF

Master of Technology

In

Metallurgical & Materials Engineering

Submitted

By

Shuvendu Tripathy

Roll No.207MM103



**Department of Metallurgical & Materials Engineering
National Institute of Technology
Rourkela
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Under the guidance of
Prof.S.Sarkar



**Department of National Metallurgical & Materials
Institute of Technology
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**National Institute of Technology
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CERTIFICATE

This is to certify that the thesis entitled, **“Studies on Aluminum–Fly-Ash Composite Produced by Impeller Mixing”**, submitted by **Shuvendu Tripathy** in partial fulfillment of the requirements for the award of Master of Technology Degree in **Metallurgical and Materials Engineering** at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under my supervision and guidance. To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/ Institute for the award of any degree or diploma.

Date:

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ABSTRACT

Metal matrix composites (MMCs) constitute an important class of design and weight-efficient structural materials that are encouraging every sphere of engineering applications. There has been an increasing interest in composites containing low density and low cost reinforcements. Among various discontinuously dispersed solids used, fly ash is one of the most inexpensive and low density reinforcement available in large quantities as solid waste by-product during combustion of coal in thermal power plants. Hence, composites with fly ash as reinforcement are likely to overcome the cost barrier for wide spread applications in automotive and small engine applications. To produce Al matrix cast particle composites, wettability of the ceramic particles by liquid Al is essential. To improve wettability, elements such as Mg and Si are added into Al melt to incorporate the ceramic particles. The present investigation has been focused on the utilization abundant available industrial waste fly ash in useful manner by dispersing it into aluminium/aluminium-magnesium/aluminium-silicon matrix to produce composites by liquid metallurgy route. Wide size range (0.1-100 μ m) fly ash particles were used. These composites were observed with the help of optical micrography, x ray micro analysis, x ray diffraction, wet chemical analysis, and image analysis. The dry sliding wear behavior of the composites in the cast conditions was studied at different loads and different sliding velocities with the help of Pin-On-Disc wear test machine. The worn surfaces and wear debris were analyzed using optical microscope and scanning electron microscope. The mechanical properties such as hardness and tensile strength have been investigated.

Introduction

Conventional monolithic materials have limitations with respect to achievable combinations of strength, stiffness, and density. In order to overcome these shortcomings and to meet the ever-increasing engineering demands of modern technology, metal matrix composites are gaining importance. In recent years, discontinuously reinforced aluminum based metal matrix composites have attracted world wide attention as a result of their potential to replace their monolithic counterparts primarily in automobile and energy sector. [1]

The basic idea is that continuous fiber reinforced composite has better strength but the processing methods is highly expensive which hinders their adoption. The continuous fiber reinforced composites do not allow secondary forming such as rolling, forging and extrusion. As results of these limitations new efforts on the research of discontinuous reinforcements have been used. [2, 3] At early stages of development of metal matrix composite emphasis was given on the preparation of fiber reinforced composite only. But due to the high cost associated with the process of production, anisotropic properties of the resultant composite and difficulties associated with the fabrication process, production of this type of composites has been limited. Now a days the particulate reinforced Al matrix composite are gaining importance because of their low cost with advantage like isotropic properties. The strengthening of aluminum alloys with dispersion of fine ceramic particulate composite materials were developed as an alternative of unreinforced alloy, for obtaining materials with high stiffness (high strength/modulus and low density) with special interest for the wear resistant and structural applications. [4, 5] The dispersion strengthened alloys can be classified, based on the size and volume % of particles uniformly dispersed in the matrix.

Aluminum alloys reinforced with ceramic particles exhibit superior mechanical properties to unreinforced Al alloys and hence are candidate for engineering applications. The aluminum metal matrix composites are produced either by casting route or by powder metallurgy. The former has the advantages of producing the composites as lower cost of production and possibility of producing larger components. However, the inherent difficulties of casting route are non wettability of ceramic particles by liquid aluminum [6], segregation of particles, higher porosity level and extensive inter-facial reaction due to higher processing temperature. Wettability of the particles can be improved by coating the particles with metals such as Ni and Cu, addition of active elements such as Mg into liquid aluminum or preheating of the

particles before addition into liquid aluminium [6]. The most conventional method of production of composites by casting route is vortex method, where the liquid aluminum containing 2-5% Mg is stirred with an impeller and ceramic particles are incorporated into vortex formed by stirring of the liquid metals. Addition of Mg into the liquid metal reduces the surface tension [7] and thereby avoids the rejection of the particles from the melts. Without addition of Mg recovery of the particles into the melt is quite low. Hence 2-5% Mg is generally added into the Al melts before incorporation of the particles. However, the chemistry of the particles of an Al alloy is changed with addition of Mg that can be deleterious to the mechanical properties of the composites.

The present investigation has been focused on utilization of waste fly ash in a useful manner by dispersing it in an aluminum matrix to produce a composite. In the present work, fly-ash which mainly consists of refractory oxides like silica, alumina, and iron oxides, was used as the reinforcing phase and to increase the wettability magnesium and silicon were added. Composites were produced with different percentages of reinforcing phase. Further, these composites were characterized with the help of, optical micrography, x-ray micro analysis, x-ray diffraction, wet chemical analysis, and image analysis. Mechanical and wear properties of the composites were also evaluated.

2. Literature Review

2.1 Definition of composite material:

The composite material can be defined as the system of material consisting of a mixture of combination of two or more micro constituents insoluble in each other and differing in form and or in material composition .These materials can be prepared by putting two or more dissimilar material in such way that they function mechanically as a single unit. The properties of such materials differ from those of their constituents. These materials may have a hard phase embedded in a soft phase or vice versa. Normally in the composite material have a hard phase in the soft ductile matrix where the hard phase act as a reinforcing agent increase the strength and modulus, and soft phase act as matrix material. The requirement for satisfying the above mentioned condition is

- a. The composite material has to be man-made
- b. The composite material must be a combination of at least two chemically distinct materials with an interface separating the components.
- c. The properties of composite should be three dimensionally combined.

2.2 Classification of Composites

2.2.1 On the basis of Matrix composite can be classified in the following groups:

a) Polymer-matrix composites (PMC)

The most common matrix materials for composites are polymeric. Polyester and vinyl esters are the most widely used and least expensive polymer resins. These matrix materials are basically used for fiber glass reinforced composites. For mutations of a large number resin provide a wide range of properties for these materials. The epoxies are more expensive and in addition to wide range of ranging commercial applications, also find use in PMCs for aerospace applications. The main disadvantages of PMCs are their low maximum working temperature high coefficients of thermal expansion and hence dimensional instability and sensitivity to radiation and moisture. The strength and stiffness are low compared with metals and ceramics. [9, 10]

b) Metal-matrix composites (MMC)

The matrix in these composites is a ductile metal. These composites can be used at higher service temperature than their base metal counterparts. These reinforcements in these materials may improve specific stiffness specific strength, abrasion resistance, creep resistance and dimensional stability. The MMCs is light in weight and resist wear and thermal distortion, so it mainly used in automobile industry. Metal matrix composites are much more expensive than PMCs and, therefore, their use is somewhat restricted. [9, 10]

c) Ceramic-matrix composites (CMC)

One of the main objectives in producing CMCs is to increase the toughness. Ceramic materials are inherently resistant to oxidation and deterioration at elevated temperatures; were it not for their disposition to brittle fracture, some of these materials would be ideal candidates for use in higher temperature and stress applications, specifically for components in automobile and air craft gas turbine engines. The developments of CMCs has lagged behind mostly for remain reason, most processing route involve higher temperature and only employed with high temperature reinforcements[9].

2.2.2 On the basis of reinforcement can be classified into three types:

a) Particle reinforced composites

Particulate reinforcements have dimensions that are approximately equal in all directions .The shape of the reinforcing particles may be spherical, cubic, platelet or any regular or irregular geometry. These composite can classified under two sub groups [8]:

- (i) Large particle composites
- (ii) Dispersion strengthened composites

b) Fiber reinforced composites

A fibrous reinforcement is characterized by its length being much greater than its cross-sectional dimension .However the ratio of length to the cross sectional dimension know as the aspect ratio, can vary considerably .In single layer composite long fibers with high aspect ratios give that are called continuous fiber reinforced composites whereas discontinuous fiber reinforced composites are fabricated using short fibres of low aspect ratio .The orientation of the discontinuous fibres may be random or preferred .The frequently encountered preferred orientation in the case of continuous fibre composite is termed unidirectional and the corresponding random situation can be approximated to by bidirectional woven reinforcement [10].

2.3 Strengthening mechanism of composite

2.3.1 Strengthening mechanism of fiber reinforced composite

Rule-Of-Mixture

Most studies concerned with the evaluation of mechanical behavior of fiber reinforced composites use what is called a "Rule-Of-Mixtures"(ROM) to predict and/or to compare the strength properties of the composite. The ROM is nothing but an operational tool that uses weighted volume average of the component properties in isolation to obtain the magnitude of the property for the composite. Specifically, in the case of composite containing uniaxially aligned, continuous fibers, the composite stress is written as

$$\sigma_c = \sigma_f V_f + \sigma_m V_m \quad (1)$$

Where σ is the axial stress, V is the volume fraction of the component and the subscripts c, f and m refer to the composite, fiber and matrix, respectively. It is to be noted that

$$V_f + V_m = 1$$

Under conditions of isostatic, i.e, the longitudinal strain in the components being equal, one may write another ROM relationship for the elastic moduli,

$$E_c = E_f v_f + E_m V_m \quad (2)$$

Where E is the elastic modulus and the subscripts represent the components as before. Eq. (2) neglects any transverse strain arising because of the different contractile tendencies of the components (i.e, $v_f = v$, where v is Poisson's ratio). However, for metallic systems, the difference in Poisson's ratio of the two components is generally insignificant and the ROM values are generally found to be within the limits of the experimental error.

Another example of a property for which ROM works very well is the density ρ . One can write as

$$\rho_c = \rho_f V_f + \rho_m V_m \quad (3)$$

It would appear from these studies that the ROM as applied conventionally to the strength properties of composites with metallic matrices is not valid. The whole is more than the sum of individual components in isolation [11].

2.3.2 Strengthening mechanism of particulate composite

a) Large-particle composite

Particle-Reinforced Composites can be large-particle and dispersion-strengthened composites are the two subclassifications of particle-reinforced composites. The distinction between these is based upon reinforcement or strengthening mechanism. The term “large” is used to indicate that particle–matrix interactions cannot be treated on the atomic or molecular level; rather, continuum mechanics is used. For most of these composites, the particulate phase is harder and stiffer than the matrix. These reinforcing particles tend to restrain movement of the matrix phase in the vicinity of each particle. In essence, the matrix transfers some of the applied stress to the particles, which bear a fraction of the load. The degree of reinforcement or improvement of mechanical behavior depends on strong bonding at the matrix–particle interface.

b) Dispersion strengthened composite

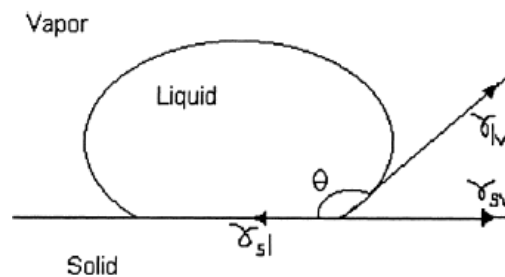
For dispersion-strengthened composites, particles are normally much smaller, with diameters between 0.01 and 0.1 μm (10 and 100 nm). Particle–matrix interactions that lead to strengthening occur on the atomic or molecular level. The mechanism of strengthening is similar to that for precipitation hardening. Whereas the matrix bears the major portion of an applied load, the small dispersed particles hinder or impede the motion of dislocations. Thus, plastic deformation is restricted such that yield and tensile strengths, as well as hardness, improve.

2.4. Interfacial bonding Mechanisms

Once the matrix has wet the reinforcement, and is therefore in intimate contact with reinforcement, bonding will occur. A number of different types of bond may be formed. The type of bonding varies from system to system and dependent on fine details such as the presence of surface contaminants or of added surface active agents.

2.4.1 Adsorption and wetting

Wettability is another significant problem when producing cast metal matrix composites. Wettability can be defined as the ability of a liquid to spread on a solid surface. Good wettability means that the liquid (matrix) will flow over the reinforcement covering every 'bump' and dip of the rough surface of the reinforcement and displacing air. It also describes the extent of intimate contact between liquid and a solid.



(Fig 2.4. Schematic diagram showing the contact angle that describes wettability)

Wettability will not occur if the viscosity of the matrix is not too high and if wetting results in a decrease in the free energy of the system. Successful incorporation of solid ceramic particles into casting requires that the melt should wet the solid ceramic phase. The problem of the wetting of the ceramic by molten metal is one of surface chemistry and surface tension. The chemistry of the particle surface, including any contamination, or oxidation, the melt surface and oxide layer must be considered. All the surfaces have associated with the free energy (per unit area)

γ_{sl} = free energy per unit area of solid- liquid interfaces, γ_{sv} = free energy per unit area of solid- vapor interfaces, γ_{lv} = free energy per unit area of liquid- vapor interfaces

The spreading coefficient can be defined as

$$SC = \gamma_{sv} - (\gamma_{sl} + \gamma_{lv}) \quad 1$$

The bonding force between the liquid and solid phases can be expressed in terms of contact angle referred to in the Young-Dupre's equation [12].

$$\gamma_{sv} = \gamma_{sl} + \gamma_{lv} \cos \theta \quad 2$$

$$\cos \theta = (\gamma_{sv} - \gamma_{sl}) / \gamma_{lv} \quad 3$$

θ may be used as a measure of the degree of wettability.

- (i) $\theta = 180^\circ$, no wettability
- (ii) $\theta = 0^\circ$, perfect wettability
- (iii) $0^\circ < \theta < 180^\circ$, partial wettability

2.4.2 Inter-diffusion

Various types of diffusion process which promote the adhesion can take place at the interface. The diffusion of free chain ends at the interface between two polymers, which leads to chain entanglements and a rise in the adhesive strength. Inter diffusion can take place in non polymeric systems, particularly if it is accompanied by a chemical reaction. The adhesive strength dependent on the nature of the resultant inters atomic bonds. Inter-diffusion plays only a minor role at low temperatures, but at elevated temperatures approaching the melting point of the matrix, inter-diffusion and chemical reaction can result in the formation of brittle inter metallics which are detrimental to the mechanical properties of MMCs.

2.4.3 Chemical bonding

Various types of chemical reaction may occur at the interface, either deliberately or inadvertent. These bonds may be covalent, ionic, metallic, etc and in many cases very strong. These are many examples of inter facial bond strength being raised by localized chemical reactions. For strong chemical bonding between the reinforcement and matrix a controlled amount of chemical reaction at the interface is always desirable. However, too thick an inter-facial zones adversely the mechanical properties of the composites and leads to premature failure.

2.4.3 Mechanical adhesion

Mechanical interlocking or keying of two surfaces can be lead to reasonable bond. Clearly the interlocking is greater and hence the mechanical bonding more effective, the rougher the interface. Also any concentration of the matrix onto the reinforcement is favorable to bonding. Mechanical bonding plays a major role in load transfer by shear. Two separate factors affect mechanical adhesion namely (1) Surface roughness, which control the amount of mechanical interlocking that can occur, and (2) The presence of residual stresses in the matrix as a result of fabrication. In most cases a purely mechanical bond is not encountered and mechanical bonding operates in conjunction with another bonding mechanism.

2.5 Metal matrix composites

Metal-matrix composites (MMCs) are engineered combinations of two or more materials (one of which is a metal) where tailored properties are achieved by systematic combinations of different constituents. Conventional monolithic materials have limitations in respect to achievable combinations of strength, stiffness and density. Engineered MMCs consisting of continuous or discontinuous fibres, whiskers, or particles in a metal achieve combinations of very high specific strength and specific modulus. Furthermore, systematic design and synthesis procedures allow unique combinations of engineering properties in composites like high elevated temperature strength, fatigue strength, damping property, electrical and thermal conductivities, friction coefficient, wear resistance and expansion coefficient. Structurally, MMCs consist of continuous or discontinuous fibres, whiskers, or particles in an alloy matrix which reinforce the matrix or provide it with requisite properties not achievable in monolithic alloys. In a broader sense, cast composites, where the volume and shape of phase is governed by phase diagrams, for example, cast iron and aluminum-silicon alloys, have been produced by foundries for a long time. The modern composites differ in the sense that any selected volume, shape and size properties in composites like high elevated temperature of reinforcement can be artificially introduced in the matrix. The modern composites are non equilibrium combinations of metals and ceramics, where there are fewer thermodynamic restrictions on the relative volume percentages, shapes and size of ceramic phases. Composite materials are attractive since they offer the possibility of attaining property combinations which are not obtained in monolithic materials and which can result in a number of significant service benefits. These could include increased strength, decreased weight, higher service temperature, improved wear resistance, higher elastic modulus, controlled coefficients of thermal expansion and improved fatigue properties. The quest for improved performance has resulted in a number of developments in the area of MMC fabrication technology. These includes both the preparation of the reinforcing phases and the development of fabrication techniques [13].

2.6 Fabrication techniques for metal matrix composites

A number of composite fabrication techniques have been developed that can be placed into four broad categories. These are: (i) powder metallurgical techniques, (ii) liquid metallurgy. The liquid metallurgy techniques include unidirectional solidifications to produce directionally aligned MMCs, suspension of reinforcement in melts followed by solidification, compocasting, squeeze casting, spray casting, and pressure infiltration. The liquid metallurgy techniques are the least expensive of all, and the multi-step diffusion bonding techniques may be the most expensive [14].

2.6.1 Powder metallurgical techniques

Powder blending followed by consolidation (PM processing), diffusion bonding and vapor deposition techniques come under solid state processing Powder metallurgy techniques offer the following three advantages over liquid metallurgy techniques for fabricating MMCs.[15]

(a) Lower temperatures can be used during preparation of a PM-based composite compared with preparation of a liquid metallurgy-based composite. The result is lesser interaction between the matrix and the reinforcement when using the' PM technique. By minimizing undesirable inter-facial reactions, improved mechanical properties are obtained.

(b) In some cases, PM techniques will permit the preparation of composites that cannot be prepared by the liquid metallurgy. For instance, fibres or particles of silicon carbide will dissolve in melts of several metals like titanium, and such composites will be difficult to prepare using liquid metallurgy techniques.

(c) However, PM techniques remain expensive compared to liquid metallurgy techniques for the composites like Al-SiC particle composites. In addition, only small and simple shape can be produced by PM techniques.

Power blending and consolidation

Blending of metallic power and ceramic fibers or particulate has the advantage of close control over the ceramic content. Blending can be carried out dry or in liquid suspension. Blending is usually followed by cold compaction, canning, degassing and high temperature consolidation stage such as hot isostatic pressing (HIP) or extrusion. PM processed AMCs, contain oxide particles in the form of plate-like particles of few tens of nm thick and in volume fractions ranging from 0-05 to 0-5 vol% depending on powder history and processing conditions. These fine oxide particles tends to act as a dispersion-strengthening agent and often has strong influence on the matrix properties particularly during heat treatment [16].

Diffusion bonding:

Mono filament-reinforced AMCs are mainly produced by the diffusion bonding (foil-fibre-foil) route or by the evaporation of relatively thick layers of aluminum on the surface of the fibre. 6061 Al-boron fibre composites have been produced by diffusion bonding via the foil-fibre-foil process [17]. However, the process is more commonly used to produce Ti based fibre reinforced composites. The process is cumbersome and obtaining high fibre volume fraction and homogeneous fibre distribution is difficult. The process is not suitable to produce complex shapes and components.

2.6.2 Physical vapor deposition:

The process involves continuous passage of fibre through a region of high partial pressure of the metal to be deposited, where condensation takes place so as to produce a relatively thick coating on the fibre. The vapor is produced by directing a high power electron beam onto the end of a solid bar feed stock. Typical deposition rates are 5–10 μm per minute. Composite fabrication is usually completed by assembling the coated fibres into a bundle or array and consolidating in a hot press or HIP operation. This technique can produce composites with uniform distribution of fibre and volume fraction as high as 80% [18].

2.6.3 Liquid metallurgy route

Liquid state processes include stir casting or compocasting, infiltration, spray casting and in situ (reactive) processing. The selection of the processing route depends on many factors including type and level of reinforcement loading and the degree of micro structural integrity desired.

Stir casting:

This involves incorporation of ceramic particulate into liquid aluminum melt and allowing the mixture to solidify. Here, the crucial thing is to create good wetting between the particulate reinforcement and the liquid aluminum alloy melt. The simplest and most commercially used technique is known as vortex technique or stir-casting technique. The vortex technique involves the introduction of pretreated ceramic particles into the vortex of molten alloy created by the rotating impeller. Lloyd (1999) has reports that vortex-mixing technique for the preparation of ceramic particle dispersed aluminum matrix composites was originally developed by Surappa & Rohatgi (1981) [19] at the Indian Institute of Science, Bangalore. Subsequently several aluminum companies further refined and modified the process which are currently employed to manufacture a variety of aluminum metal matrix composites on commercial scale [20].

The vortex method is one of the better known approaches used to create and maintain a good distribution of the reinforcement material in the matrix alloy. In this method, after the matrix material is melted, it is stirred vigorously to form a vortex at the surface of the melt, and the reinforcement material is then introduced at the side of the vortex. The stirring is continued for a few minutes before the slurry is cast. There are different designs of mechanical stirrers. Among them, the turbine stirrer is quite popular. During stir casting for the synthesis of composites, stirring helps in two ways: (a) transferring particles into the liquid metal, and (b) maintaining the particles in a state of suspension.

Micro structural inhomogeneities can cause notably particle agglomeration and sedimentation in the melt and subsequently during solidification. Inhomogeneity in reinforcement distribution in these cast composites could also be a problem as a result of interaction between suspended ceramic particles and moving solid-liquid interface during solidification. Generally it is possible to incorporate up to 30% ceramic particles in the size range 5 to 100 μm in a variety of molten aluminum alloys. The process is not suitable for the incorporation of sub-micron size ceramic particles or whiskers. Another variant of stir casting process is compocasting. Here, ceramic particles are incorporated into the alloy in the semi solid state.

Infiltration process:

Liquid metal is injected into the interstices of the porous preforms of continuous fibre/short fibre or whisker or particle to produce MMCs. Depending on the nature of reinforcement and its volume fraction preform can be infiltrated, with or without the application of pressure or vacuum. MMCs having reinforcement volume fraction ranging from 10 to 70% can be produced using a variety of infiltration techniques. In order for the preform to retain its integrity and shape, it is often necessary to use silica and alumina based mixtures as binder. Some level of porosity and local variations in the volume fractions of the reinforcement are often noticed in the MMCs processed by infiltration technique. The process is widely used to produce aluminum matrix composites having particle/whisker/short fibre/continuous fibre as reinforcement.

In most of the cases the fibres do not act as preferential crystal nucleation sites during melt solidification. One consequences of this is that the last liquid to freeze, which is normally solute enriched, tend to locate around the fibres[21].

Spray deposition:

Spray deposition techniques fall into two distinct classes, depending whether the droplet stream is produced from a molten bath (Osprey process) or by continuous feeding of cold metal into a zone of rapid heat injection (thermal spray process). The spray process has been extensively explored for the production of MMCs by injecting ceramic particle/whisker/short fibre into the spray. MMCs produced in this way often exhibit inhomogeneous distribution of ceramic particles. Porosity in the as sprayed state is typically about 5–10%. Depositions of this type are typically consolidated to full density by subsequent processing. Spray process also permit the production of continuous fibre reinforced aluminum matrix composites. For this, fibres are wrapped around a mandrel with controlled inter fibre spacing, and the matrix metal is sprayed onto the fibres. A composite mono type is thus formed; bulk composites are formed by hot pressing of composite mono types. Fibre volume fraction and distribution is controlled by adjusting the fibre spacing and the number of fibre layers. MMCs processed by spray deposition technique are relatively inexpensive with cost that is usually intermediate between stir cast and PM processes.

In-situ processing (reactive processing):

There are several different processes that would fall under this category including liquid-gas, liquid-solid, liquid-liquid and mixed salt reactions. In these processes refractory reinforcement are created in the aluminium alloy matrix. One of the examples is directional oxidation of aluminium also known as DIMOX process. In this process the alloy of Al–Mg is placed on the top of ceramic preform in a crucible. The entire assembly is heated to a suitable temperature in the atmosphere of free flowing nitrogen bearing gas mixture. Al–Mg alloy soon after melting infiltrates into the preform and composite is formed.

Martin–Marietta’s exothermic dispersion process or the XDTm process is another in-situ technique for composite processing. XDTm process is used to produce TiB₂ reinforced aluminium matrix composites. The process is flexible and permits formation of both hard and soft phases of various sizes and morphologies that includes whiskers, particles and platelets in aluminium alloy matrices.

Gas–liquid reaction is also utilized to produce TiC reinforced aluminium matrix composites. For example, by bubbling carbonaceous gas like methane into Al–Ti melt kept at elevated temperature it is possible to produce Al–TiCp composites [22]. London and Scandinavian Metallurgical Company has developed an in-situ technique, which utilizes reaction between

mixed salts to produce a dispersion of fine TiB_2 particles in an aluminium matrix. A major limitation of in-situ technique is related to the thermodynamic restrictions on the composition and nature of the reinforcement phase that can form in a given system, and the kinetic restrictions on the shape, size and volume fraction of the reinforcement that can be achieved through chemical reactions under a given set of test conditions.

2.7 Role of magnesium in cast aluminium alloy matrix composites

Wettability is a most significant problem when producing cast metal matrix composites. Particle –matrix interface have important effect on the mechanical properties of the composites, as good bonding promote load transfer to the higher strength ceramic particles. On the other hands, extensive inter facial reactions may deteriorate the mechanical properties of the composites. B. C. Pai et al. [23] reported that magnesium played an important role during the synthesis of aluminum alloy matrix composites with dis-persoids such as zircon (ZrSiO_4), zirconia (ZrO_2), titania (TiO_2), silica (SiO_2), graphite, aluminium oxide (Al_2O_3) and silicon carbide (SiC). Magnesium is one of the important alloying elements in aluminium. In Al-Si casting alloys Mg_2Si is the key phase for alloy strengthening. Magnesium addition to aluminium reduces its casting fluidity at the same time as it reduces the surface tension of the aluminium sharply [24].

The presence of magnesium in aluminium alloy matrix during composite fabrication not only strengthens the matrix but also scavenges the oxygen from the surface of the dis-persoid, leading to an increase in the surface energy, of the dis-persoids. It can reduce Al_2O_3 , either to form Al, MgAl_2O_4 or MgO depending upon its concentration. Hence there are three important role of magnesium in aluminium melt.

- Increasing the surface energies of the ceramics
- Decreasing the surface tension of the liquid matrix alloy
- Decreasing the solid/liquid interracial energy at the dispersoid matrix interface.

2.8 Particulate reinforced metal matrix composites

The other tendency in the research of Al matrix composites is to develop more inexpensive production techniques especially for discontinuously reinforced MMCs. The large consumption of monolithic aluminium alloys has enabled the development of production technology to very efficient and reliable level. Many of these technologies can be applied to production of aluminium matrix composites as well. For instance, standard shaping methods, such as extrusion, forging and rolling, can be used. Traditionally, liquid state processes [25-26, 6], such as various casting methods, and powder metallurgical methods are used in production of particulate reinforced Al matrix composites. However, new deposition and in situ processes are potentially very efficient and economical and, therefore, they are actively studied. Powder metallurgical route is difficult to be automated and, thus it is very probably not the right answer for economical production of Al matrix composites. Hence, the most promising processes are found among on the liquid state, deposition and in situ processes. The most simple, inexpensive and widely used methods for monolithic Al parts are various casting techniques. It is therefore natural that a lot of emphasize is put on developing these techniques for Al matrix composites as well. In order to ensure homogenizes distribution of reinforcement particles in the matrix metal stirring by using mechanical, electromagnetic or gas mixing is necessary. Especially, semi-solid slurry mixing methods, such as compocasting or rheocasting [27], have gathered increasing interest. Because of the high viscosity of the slurry higher volume fractions of reinforcement can be processed than in normal casting and the particles are more homogeneously distributed as well. It is even possible to use non-wetting particles, which are entrapped in the slurry. Moreover, the reactions between the matrix and reinforcements are reduced.

The SiC particles are the most common discontinuous reinforcements in Al matrix composites although the density of SiC is slightly higher than that of Al. This is because it is inexpensive and readily available but still gives the composite high strength and elastic modulus. The improved wear resistance is often the primary feature as well. In the same way as in the case of continuous SiC fibres the possibility of chemical reactions limits the high temperature applications and may cause problems in production. Excess Si reduces the reactivity of SiC in Al remarkably. Another widely used particulate reinforcement in Al matrix composites is Al_2O_3 . In comparison to SiC it is much more inert in Al and it is also oxidation resistant. Accordingly, it is much more suitable for high temperature fabrication and use. In order to overcome the problem of poor wettability of Al_2O_3 by Al, which disturbs

especially the liquid stirring production routes, the matrix is alloyed or the reinforcement is surface coated. As described previously, Li is found to be a beneficial alloying element. MgO on the surfaces of Al_2O_3 is also improving the wettability. On the other hand, in squeeze casting the wettability is not as large problem as in liquid stirring. Therefore, this efficient technique has been often utilized in production of particulate Al_2O_3 reinforced Al matrix composites. In fact, this combination of production method and composite material Al is currently the most promising candidate for large scale production of relatively inexpensive MMCs for general Al applications. In the early stage of development of the cast particulate composite, the particles are injected in molten Al through carrier gas[28], to achieve greater recovery, the coating of the particles has been suggested than the uncoated one e.g. Nickel coating with graphite particles in case of Al matrix composites. Ni improves the wettability of the particle by Al melt. Not only Ni but also the addition of Mg, Li, Si Ca, in Al melt improves wettability either by changing the interface energy through some interracial reaction or by modifying the oxide layers in the metal surface Detailed investigation of Pai et al. and P.K.Rohatgi [6] and co workers have observed that magnesium increases the wettability of the fly ash in the Al melt. In a recent study it has been demonstrated that the addition of Ca improves the wettability of Al 4.5% eu melt by its effects on improvement of retention of particles is less that with Mg addition. To ensure the uniform distribution of particles, stirring of the melt are suggested.

To ensure the uniform distribution of particles, stirring of the melt are suggested. Pellet methods are developed to incorporate ceramic particles into Al melt by Pai and Mc Donald and co workers [29, 30]. In this method the coarser particles of base alloy and reinforcing ceramic particles are mixed and pressed to form pellets. These pellets were subsequently plunged into the melt followed by slow stirring manually or mechanically. But the distribution of particles of the cast particulate composite was not satisfactory uniform when it is prepared by injection technique or pallet method without stirring. To over come such difficulty Vortex method was developed [19, 31]. This the most common method used at present for cast metal matrix particulate composite. In this method molten metal in a crucible is stirred with a available impeller to form a vortex in the melt. Required amount of ceramic particles are added into the vortex at a predetermined rate. The particles get entrapped in the melt and distribute uniformly in it during stirring. The development of SiO_2 coating on fly ash particles is demonstrated as an effective way to improve matrix-reinforcement bonding during the manufacturing by a liquid route of Al matrix composite [32].

Heat treatment of the particles before dispersion into the melt aids their transfer by causing desorption of adsorbed gases from the particle surface. Heating silicon carbide particles to 900°C, for example, assists in removing surface impurities and in the desorption of gases, and alters the surface composition by forming an oxide layer on the surface [33]. The addition of pre-heated alumina particles in Al-Mg melt has been found to improve the wetting of alumina [34]. A clean surface provides a better opportunity for melt-particles interaction, and thus, enhances wetting. Ultrasonic techniques, various etching techniques, and heating in a suitable atmosphere can be used to clean the particle surface. Ultrasonic vibration has been applied to molten aluminium in order to improve the wettability of alumina particles [35].

S. Sarkar and S. C. Panigrahi have worked on the Effect of Particle Distribution on the properties of Aluminum Matrix In-situ Particulate Composites [36] and conclude that discontinuously reinforced aluminum matrix composites are fast emerging materials that compete with conventional metallic materials. In-situ particulate composites in comparison with conventional cast particulate composites produced by external addition, promote cleaner interfaces, eliminate interface incompatibility of the matrices with the reinforcements, help to achieve greater thermodynamic stability of reinforcement particles in the matrix at elevated temperature, and also increase the possibility of developing coherency between the matrix and particles formed in-situ. The morphology and the distribution of particles strongly influence the physical and mechanical properties of the composites. In the present study, ilmenite was added to molten aluminum, aluminum–magnesium and aluminum–silicon alloys by vortex method. The oxides present in ilmenite are observed to react with aluminum, magnesium and resulting in production of Al_2O_3 , MgO and metallic Fe and Ti, which dissolved in liquid aluminum. The strength and hardness value showed considerable improvement. The resulting composites also show appreciable ductility

2.9 Aluminium -fly ash particulate reinforced composite

M. Ramachandra K. Radhakrishna [37] has worked on the Effect of reinforcement of fly ash on sliding wear, slurry erosive wear and corrosive behavior of aluminium matrix composite. Al (12 wt% Si) as matrix material and up to 15 wt% of fly ash particulate composite was fabricated using the stir casting rote and came forward into following conclusions

- Fly ash improves abrasive wear resistance (20-30%) of Al. and reduces the coefficient of friction.
- Increase in normal load and sliding velocity increases magnitude of wear and frictional force.
- Different wear mechanisms were studied under varying different parameter such as normal load, % of fly ash content and sliding velocity. Four different mechanisms are found that are abrasion, oxidation, delamination, thermal softening and adhesion.
- Corrosion resistance of reinforced composite has decreased with increase in flyash content.

Sudarshan, M.K. Surappa [38, 39] have synthesized A356 Al-fly ash particle composites .They studied mechanical properties and dry sliding wear and come into brief idea that

- Fly ash with narrow size range (53–106 μ m) show better properties compared with the wider size range (0.5–400 μ m) particles.
- The damping capacity of composite increases with the increase in volume fraction of fly ash.
- Fracture surface of composites show mixed mode (ductile and brittle) fracture.
- The 6% of fly ash particles into A356 Al alloy shows low wear rates at low loads (10 and 20 N) while 12% of fly ash reinforced composites show lower wear rates compared to the unreinforced alloy in the load range 20–80 N.
- The types of wear dominant in unreinforced alloy are adhesive wear, whereas abrasive wear is predominant in composites. At higher load, subsurface delamination is the main mechanism in both the alloy as well in composites.

R.Q. Guoa and P.K. Rohatgi, while studding the changes of chemical reaction between the Al and the fly ash during synthesis or reheating [40] of fly ash found out that

- The chemical reactions do occur between the fly ash an Al melts. The SiO₂ and Fe₂O₃ present in the fly ash is reduced to Si and Fe .The melt oxidized to Al₂O₃.

- The above mentioned reactions do occur rapidly at the temperature more than 850⁰C .At this higher temperature the free energy of transformation has significant negative value to occur this reaction spontaneously.
- The reaction between Al and fly ash complete after 10 hours

P. K. Rohatgia, J. K. Kima, [41] and co workers have worked on Compressive characteristics of A356/fly ash cenosphere composites synthesized by pressure infiltration technique and conclude that

- Using gas pressure infiltration up to 20 to 65% volume percentage of fly ash can be reinforced.
- The various factors affecting the densities of the composites melt temperature, applied pressure, and the size of particles.

P. K. Rohatgi, N. Gupta, and Somon Alaraj [42] have studied the coefficient of thermal expansion of pure Al containing 65 vol% of hollow fly ash particles and suggested that

- Composites with a lower coefficient of thermal expansion can be made by incorporating cenospheres under controlling the processing parameter for a given volume fraction of reinforcement.
- Composite synthesized at different pressure for different infiltration time (min) and came to conclude that increase in the infiltration pressure and temperature improves the infiltration and decreases the entrapped air voids, as a result of which lower the coefficient of thermal expansion.

J. Sobczak, J. Bienias and co workers [43] have synthesized fly ash particles as reinforcement in metal matrix composites by squeeze casting technology and gravity casting and compared between them and have come to following conclusion

- Squeeze casting technology is much useful in comparison with gravity casting. Because it has structural homogeneity with less porosity and good interracial bonding.
- Due to presence of fly ash particle in the composite the corrosion is pitting type corrosion.

S. Sarkar, S. sen and S. C. Mishra and co workers has studied on Aluminum – fly ash composite produced by impeller mixing[7] and came into a brief idea that

- Up to 17wt% fly ash reinforcement can be reinforced by liquid metallurgy route.

- The addition of magnesium into the aluminium melt increase the wettability and thus increase in the mechanical properties such as hardness, tensile strength and the wear resistance is observed.

P.K. Rohatgi, D. Weiss, and Nikhil Gupta [44] in this paper studied that al- fly composite can be used for automotive and other applications and show details on environmental and energy benefits.

- The potential cost, energy, and pollution savings as a result of incorporation of fly ash in aluminum is huge.
- The potential reduction in cost and energy content of individual auto parts, energy consumption, and emissions due to the replacement of 20% aluminum by fly ash show the substantial benefit of using ALFA composites.

2.10 FLY ASH

Fly ash is one of the residues generated in the combustion of coal. It is an industrial byproduct recovered from the flue gas of coal burning electric power plants. Depending upon the source and makeup of the coal being burned, the components of the fly ash produced vary considerably, but all fly ash includes substantial amounts of silica (silicon dioxide, SiO_2) (both amorphous and crystalline) and lime (calcium oxide, CaO). In general, fly ash consists of SiO_2 , Al_2O_3 , and Fe_2O_3 as major constituents and oxides of Mg, Ca, Na, K etc. as minor constituent. Fly ash particles are mostly spherical in shape and range from less than $1\text{ }\mu\text{m}$ to $100\text{ }\mu\text{m}$ with a specific surface area, typically between 250 and $600\text{m}^2/\text{kg}$. The specific gravity of fly ash vary in the range of 0.6 - 2.8 gm/cc . Coal fly ash has many uses including as a cement additive, in masonry blocks, as a concrete admixture, as a material in lightweight alloys, as a concrete aggregate, in flow able fill materials, in roadway/runway construction, in structural fill materials, as roofing granules, and in grouting. The largest application of fly ash is in the cement and concrete industry, though, creative new uses for fly ash are being actively sought like use of fly ash for the fabrication of MMCs.

2.10.1 Classification on the basis of Chemical Composition:

Fly ash is a pozzolanic material and has been classified into two classes, F and C, based on the chemical composition of the fly ash. According to ASTM C 618, the chemical requirements to classify any fly ash are shown in Table 2.10.1.

Table 2.10.1 Chemical Requirements for Fly Ash Classification

Properties	Class F	Class C
Silicon dioxide (SiO_2) plus aluminum oxide (Al_2O_3) plus iron oxide (Fe_2O_3), min, %	70.0	50.0
Sulfur trioxide (SO_3), max, %	5.0	5.0
Moisture Content, max, %	3.0	3.0
Loss on ignition, max, %	6.0*	6.0

2.11.2 On basis of size, shape and structure:

1. Precipitator fly ash

It is spherical in nature, the spheres are solid and the density is in the range of $2.0\text{--}2.5\text{ g cm}^{-3}$.

2. Cenosphere fly ash

It is also spherical in shape but these spheres are hollow, so the density of this kind of fly ash is very less as compared to the precipitator fly ash. Here density is less than 1 gm cm^{-3} ($0.3\text{--}0.6\text{ gm/cc}$)

2.11 WHY FLY ASH

1. The preference to use fly ash as a filler or reinforcement in metal and polymer matrices is that fly ash is a byproduct of coal combustion, available in very large quantities at very low costs since much of this is currently land filled. Currently, the use of manufactured glass micro spheres has limited applications due mainly to their high cost of production. Therefore, the material costs of composites can be reduced significantly by incorporating fly ash into the matrices of polymers and metallic alloys. However, very little information is available on to aid in the design of composite materials, even though attempts have been made to incorporate fly ash in both polymer and metal matrices. Cenosphere fly ash has a lower density than talc and calcium carbonate, but slightly higher than hollow glass. The cost of cenosphere is likely to be much lower than hollow glass. Cenosphere may turn out to be one of the lowest cost fillers in terms of the cost per volume.

2. The high electrical resistivity, low thermal conductivity and low density of fly-ash may be helpful for making a light weight insulating composites.

3. Fly ash as a filler in Al casting reduces cost, decreases density and increase hardness, stiffness, wear and abrasion resistance. It also improves the maintainability, damping capacity, coefficient of friction etc. which are needed in various industries like automotive etc.

4. As the production of Al is reduced by the utilization of fly ash. This reduces the generation of green house gases as they are produced during the bauxite processing and alumina reduction.

2.12Chemical reaction between al and fly ash

The thermodynamic analysis indicates that there is possibility between the reaction of Al melt and the fly ash particles. The particles contain alumina, silica and iron oxide which during solidification process of Al fly ash composites or during holding such composites at temperature above 850⁰ C, are likely to undergo chemical reactions, reported by P.K.Rohatagi and Guo. The experiments indicate that there is a progressive reduction between SiO₂, Fe₂O₃ and mullite by Al and formation of Al₂O₃, Fe and Si. The wall of cenosphere fly ash particles progressive disintegrates into discrete particles into the reaction progress.

2.13 Wear behavior

Wear behavior is the surface damage or removal of material from one or both of two solid surfaces in a sliding, rolling, or impact motions relative to one another. So it is surface phenomenon that occurs by displacements and detachments of materials. Wear problems generally differ from those entailing outright breakage, as wear usually a progressive loss of weight and alterations of dimensions over a periods of time.

Wear is undesirable products in almost all machine applications such as bearings seals gears, and cams etc. Wear of those components may range forms mild polishing type attrition to rapid and severe removal of material accommodating with surface roughing. Whether or not wear constitutes failure of these components depends upon whether the wear deleteriously affects the ability of the components to function. Even mild polishing type wear of a close fitting pool in a hydraulic valve may cause excessive leakage and thus constitute failure, even though the surface of the pool is smooth and apparently undamaged. On the other hand, a hammer in rock crusher can continue to function satisfactorily in spite of severe detecting, gouging and removal of as much as several inches of surface metal.

Laws of wear

The laws governing wear are not completely clear but both adhesive and abrasive wear a simple equation can be written as:

$$V/X = K*(W/H)$$

Where,

V=Wear volume

X=Sliding distance

W=Normal load applied

H=Initial hardness of the softer sliding components

K= Wear coefficients

Further it has also been shown that there is relation between hardness H of the metal and its yield or flow pressure σ_y which given by:

$$H= 3* \sigma_y$$

From these equations we can conclude that the total volume of the metal removed due to sliding is directly proportional to applied loads, sliding distance and inversly proportional to hardness of the softer sliding component.

2.12.1Types of wear

Wear has been categorized in various ways. The phenomenological approach is based on a macroscopic description of appearance of the worn surface. For that is suffering, rubbing fitting. But his system has its limitations as it goes not focus on his mechanism of wear and therefore must relay almost entirely on imperial solutions to wear problems.

Another way is to categorize wear ways on the fundamental mechanism that operating but this approach is complicated by the fact that more than one mechanism may be operating at a time and by the lack of sufficient information .A third type of classification describes wear based on the shape and size of the wear debris particles generated.

A potential wear situ tons exists whenever there is a relative motion between two solids under loads. Broadly speaking the motion can be unit directional or reciprocating either sliding or rolling .There may be a combination of rolling and sliding are wear may occur due to oscillatory movement at small amplitudes metal can interact with non metal or liquids such as lubricating or marine water.

Depending on the nature of movement of the media following types wear has been classified.

- Abrasive wear
- Adhesive wear
- Corrosion wear
- Erosive wear
- Fatigue wear
- Fretting

Abrasive wear

Abrasive wear occurs when asperities of rough, hard surface or hard particles slide on a softer surface and damage the interface by plastic deformation or fracture. In case of ductile materials with high fracture toughness, hard asperities or hard particles result in the plastic flow of the softer material. Most metallic and ceramic surfaces during sliding show clear evidence of plastic flow, even some ceramic brittle materials. In one way abrasive wear is classified as gouging abrasion, high stress (grinding) abrasion. In` gouging, abrasion large particles are removed from surface, leaving dip groves and/or pits .High stress or grinding abrasion is accompanied by fracture of the abrasive particles. Low stress or scratching abrasion occurs when the loads is low enough that the abrasive particles are not fracture.

Another way of classification divides abrasion into two bodies or three body abrasion. In the first case the hard surface is the harder of two rubbing surface (two body abrasion), for

example, in mechanical operations such as grinding, cutting and machining. In three body abrasion; the hard surface is a third body generally a small particle of abrasive, caught between the two surfaces and sufficiently harder that is able to abrade either one or both mating surfaces, for examples, in free-abrasive lapping and polishing.

Adhesive wear

Adhesive wear occurs when two nominally flat bodies are in sliding contact, whether lubricated or not. Adhesion (or bonding) occurs at the asperity contacts at the interface, and these contacts are shared by sliding which may result in detachment of a fragment from one surface and attachment to the other surface .As the sliding continues the transferred fragment may come off the surface on which they are transferred and be transferred back to original surface ,or else from loses wear particles .Some are fractured by fatigue process during repeated loading and unloading action resulting in the form of lose particles.

Corrosive wear

Wear where contribution to the wear rate by the chemical reaction with the environment .In certain cases chemical reaction is followed by removal of the corrosion product by mechanical action that is abrasion .It may be occur that the mechanical action precedes the chemical action and results in he formation of very small particles of debris, which subsequently react with environment. Due to the chemical reaction change the corrosion by reaction with environment.

Erosive wear

This type of wear results when grits impinge on solids while cavitations erosion may arise when a components rotates in a medium .Erosion of surface can take place in a liquid medium,even without the presence of solid abrasive in that medium. Cavitations involve formation and subsequent collision of bubbles within the liquids .The collision of liquid droplets with the solid surface at high speed results in form of liquid erosion called liquid impingement. It involves the progressive removal of materials from surfaces by repeated impulse loading at microscopically small areas.

Fatigue wear

Fatigue wear arises as a result of cyclic loadings for examples in rolling elements bearings lose of materials occurs by spelling of surface layers .It may be due to failure of lubricated contacts as in gears, friction drives and ball and roller bearings. The fatigue cracks are believed starts below the surface at a point where the shear stress is maximum so improve

the components .Working is done at low contacts loads and having an optimum depth of hard cases coupled with a high degree of surface finish .

Fretting

Fretting occurs where low amplitude oscillatory motion in tangential direction takes place between contacting surfaces, which are nominally at rest. This is a common occurrence since most machinery is subjected to vibration, both in transit and in operation .Basically, fretting is a form of adhesive or abrasive wear where the normal load causes adhesion between asperities and oscillatory movement causes rupture resulting in wear debris.

Fretting may be of two type; One is contact collision that takes place between the bore of bearings and the shaft or between the outside surface of bearing contact and bore housing.

Another type is fretting damage within bearing contact area .This type of fretting is frequently referred as false braining.

3. EXPERIMENTAL WORK

3.1 Raw materials

The matrix material used in the experiment investigation was commercially pure aluminium. The fly ash was collected from RSP steel plant Orissa, India. The particle size of the fly ash received condition lies in the range from (0.1-100 μm).

3.2 Melting and casting

The aluminum fly ash metal matrix composite was prepared by stir casting route. For this we took 500gm of commercially pure aluminum and desired amount of fly ash particles. The fly ash particle was preheated to 300⁰C for three hour to remove moisture. Commercially pure aluminum was melted in a resistance furnace. The melt temperature was raised up to 720⁰C and it was degassed by purging hexa chloroethane tablets. Then the melt was stirred with the help of a mild steel turbine stirrer. The stirring was maintained between 5 to 7 min at an impeller speed of 200 rpm. The melt temperature was maintained 700⁰C during addition of fly ash particles. The dispersion of fly ash particles were achieved by the vortex method. The melt with reinforced particulates were poured into the preheated permanent metallic mold. The pouring temperature was maintained at 680⁰C. The melt was then allow to solidify the moulds. The composites were made with a different amount of fly-ash (i.e. 5, 10, 20, wt %),

Magnesium and silicon were added to increase the wettability of fly ash particles.

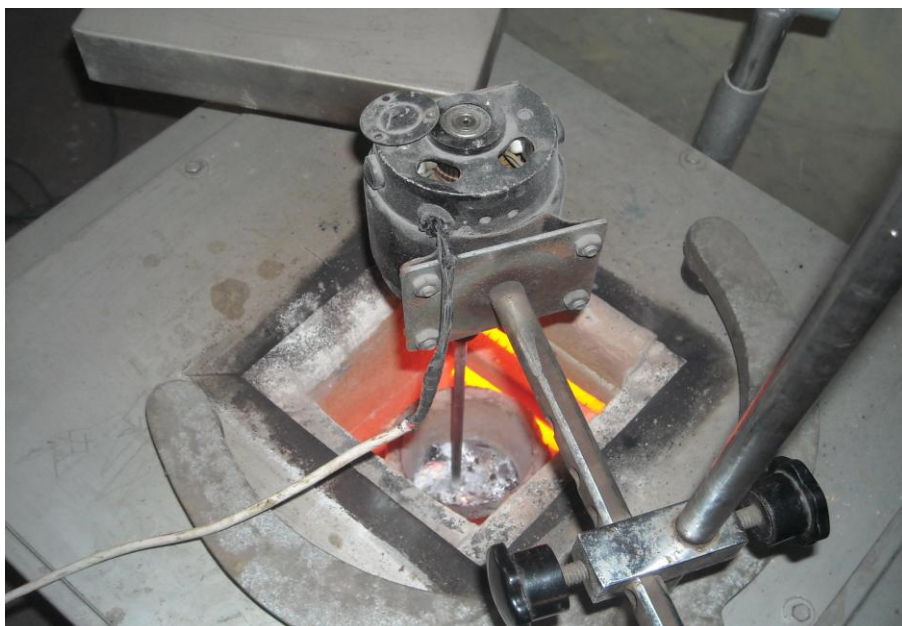


Fig 3.1.Laboratory stir casting set up

3.3 Microstructural characterization.

3.3.1 Scanning electron microscopy

Micro structural characterization studies were conducted on unreinforced and reinforced samples. This is accomplished by using scanning electron microscope. The composite samples were metallographically polished prior to examination. Characterization is done in etched conditions. Etching was accomplished using Keller's reagent. The SEM micrographs of composite and wear debris were obtained using the scanning electron microscope. The images were taken in both secondary electron (SE) and back scattered electron (BSE) mode according to requirement. Microscopic studies to examine the morphology, particle size and micro structure were done by a JEOL 6480 LV scanning electron microscope (SEM) equipped with an energy dispersive X-ray (EDX) detector of Oxford data reference system. Micrographs are taken at suitable accelerating voltages for the best possible resolution using the secondary electron imaging.



Fig 3.2: JEOL JSM-6480LV scanning electron microscope

3.3.2 Optical microscopy

The casting procedure was examined under the optical microscope to determine the cast structure. A section was cut from the castings. It is first belt grinded followed by polishing with different grade of emery papers. Then they were washed and polished in clothes and then washed, dried and etched with Keller's solution and then examined through optical microscope.

3.4 Particle size analysis

Particle size of the milled powder was measured by Malvern particle size analyzer (Model Micro-P, range 0.05-550 micron). Firstly, the liquid dispersant containing 500 ml of distilled water was kept in the sample holder. Then the instrument was run keeping ultrasonic displacement at 10.00 micron and pump speed 1800 rpm.



Figure 3.3: Malvern particle size analyzer (Model Micro-P, range 0.05-550 micron).

3.5 Mechanical properties observation.

3.4.1 Hardness

Bulk hardness measurements were carried out on the base metal and composite samples by using standard Brinnel hardness test. Brinnel hardness measurements were carried out in order to investigate the influence of particulate weight fraction on the matrix hardness. Load applied was 750kgs and indenter was a steel ball of 5 mm diameter.

3.4.1 Tensile test

The tensile testing of the composite was done, on Instron testing machine. The sample rate was 9.103pts/sec and cross-head speed 5.0 mm/min. Standard specimens with 30mm gage length were used to evaluate ultimate tensile strength. The comparison of the properties of the composite material was made with the commercially pure Al.

3.6 Sliding wear behavior

Wear has been defined as the displacement of material caused by hard particles or hard protuberances where these hard particles are forced against and moving along a solid surface. Two body sliding wear tests were carried out on prepared composite specimens. A Ducom, Bangalore makes computerized pinion- disc wear test machine was used for these tests. The wear testing was carried out at a constant sliding velocity of 1m/sec with normal loads of 10N, 15N, 20N. A cylindrical pin of size 1.1cm diameter and 2.1cm length prepared from composite casting was loaded through a vertical specimen holder against horizontal rotating disc. Before testing, the flat surface of the specimens was abraded by using 2000 grit paper. The rotating disc was made of carbon steel of diameter 50 mm and hardness of 64 HRC. Wear tests were carried out at room temperature without lubrication for 30 min. The principal objective of investigation was to study the coefficient of friction and wear.

3.7 Worn surface and debris examination

The debris and worn surface wear pin were studied under SEM and optical microscope.

4. Results and discussions

4.1 Fly ash analysis

Table 4.1 Composition of fly ash used as reinforcement in wt%

Compounds	Percentages (%)
SiO ₂	67.2
Al ₂ O ₃	29.6
Fe ₂ O ₃	0.1
CaO	1.4
MgO	1.7

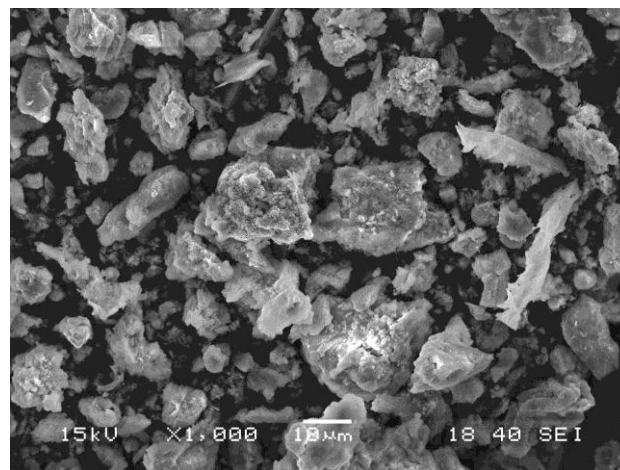


Fig 4.1 SEM Micrograph of fly ash used in the study.

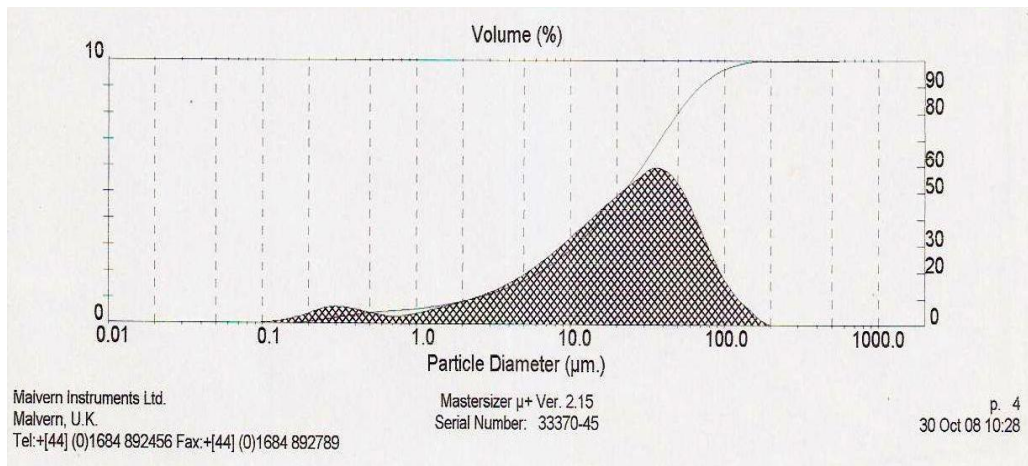


Fig.4.2- Particle size analysis of fly ash

Fly ash from Rourkel steel plant (India) had a wide particle size distribution. The particle size of the fly ash as received condition, lies in the range from (0.1-100 μm). The SEM micro-graph of the fly ash is shown in fig4.1. The major components of fly ash as received from the source and used for reinforcement are listed in Table 4.1 in wt%. The fly ash consists mainly Al_2O_3 (29.6 wt %) and SiO_2 (67.2wt %).

4.2. Optical microstructure of cast composites

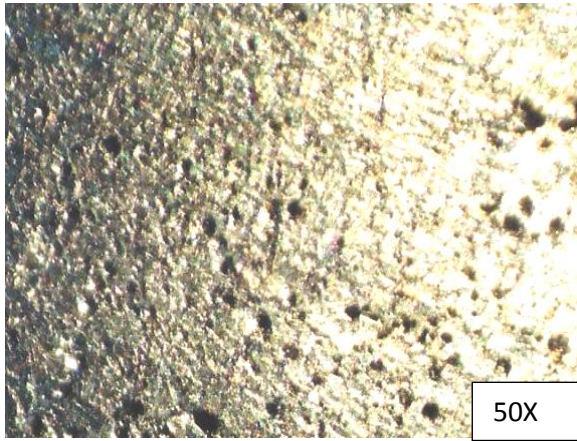


Fig.4.3- Al+ 5% fly ash composite .

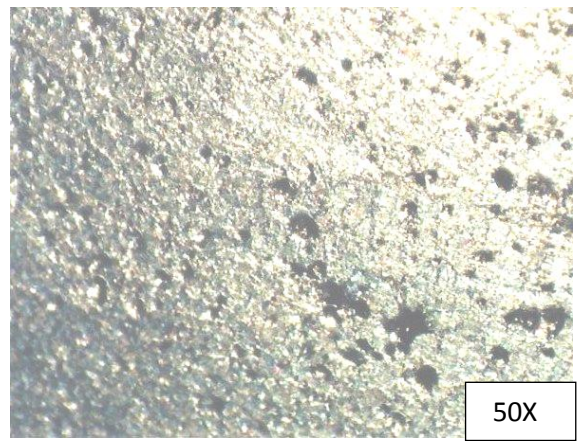


Fig4.4- Al+ 10% fly ash composite

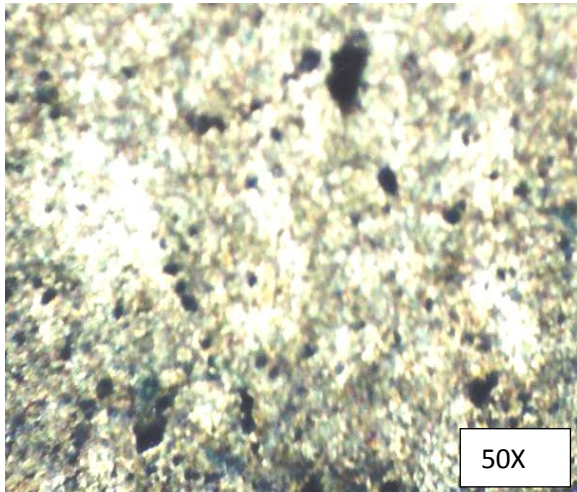


Fig.4.5-Al+15% fly ash composite

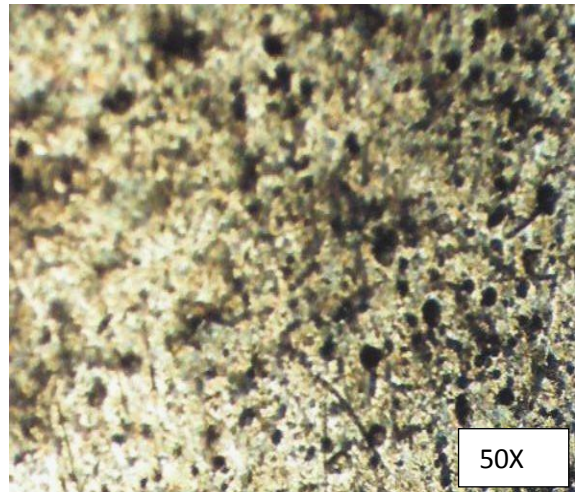


Fig.4.6-Al+20% fly ash composite

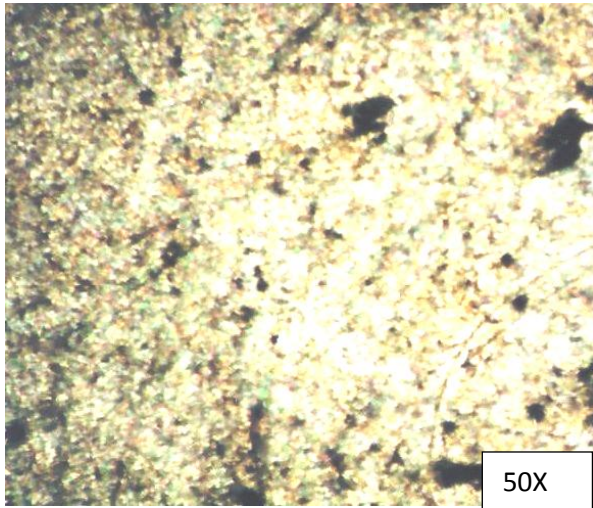


Fig.4.7-Al+2%Si+10% fly ash composite

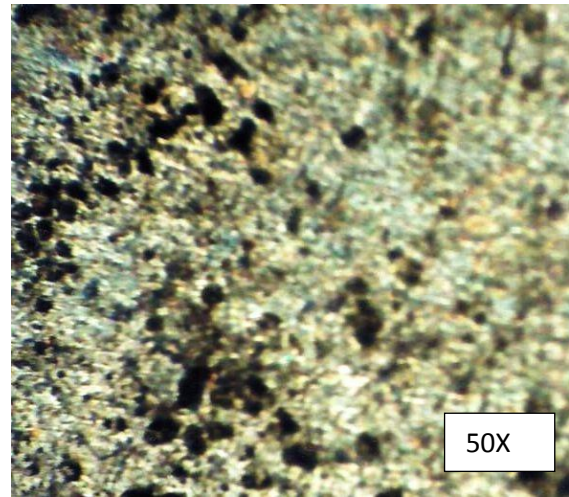


Fig.4.8- Al+2%Mg+10% fly ash composite

4.3. SEM micrographs of the MMCs

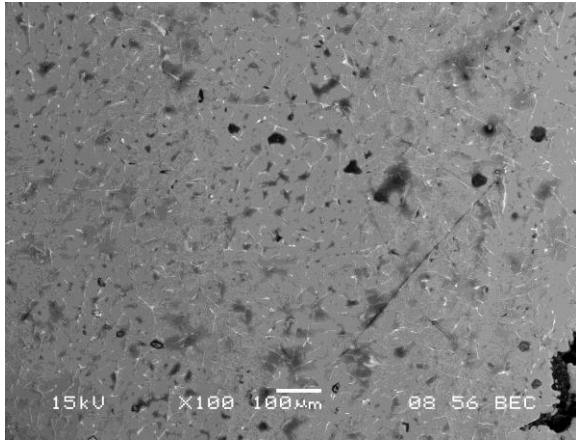


Fig.4.9- Al+ 5% fly ash composite

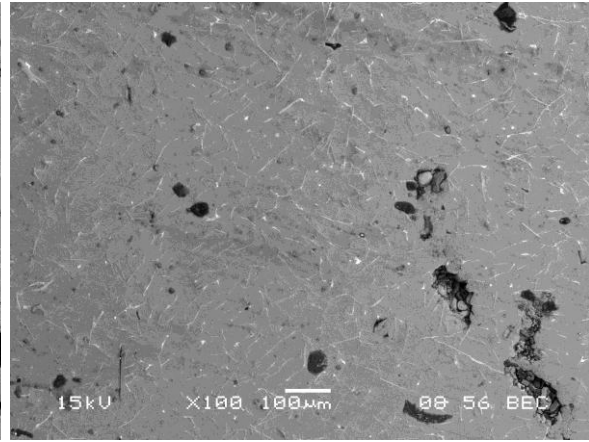


Fig.4.10- Al+ 10% fly ash composite

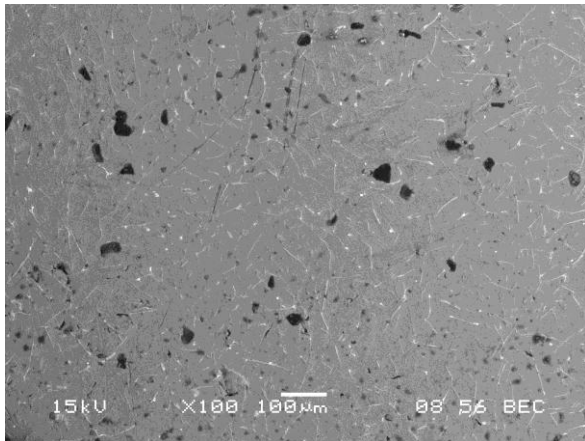


Fig.4.11- Al+ 15% fly ash composite

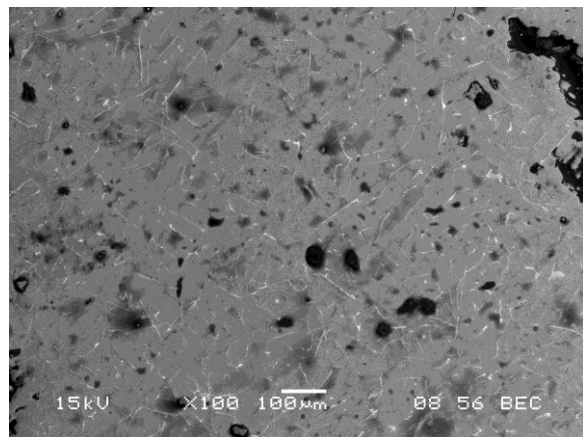


Fig.4.12- Al+ 20% fly ash composite

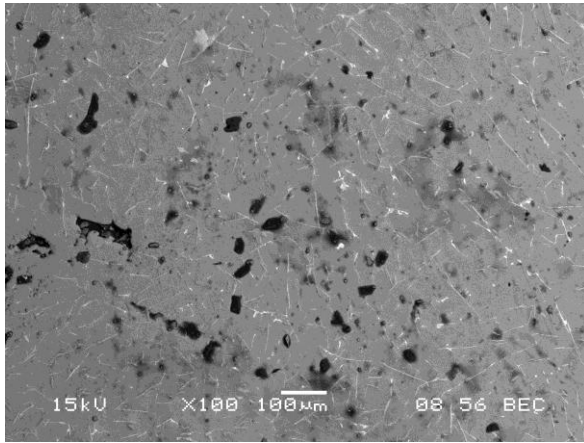


Fig.4.13- Al+2%Si+10% fly ash composite

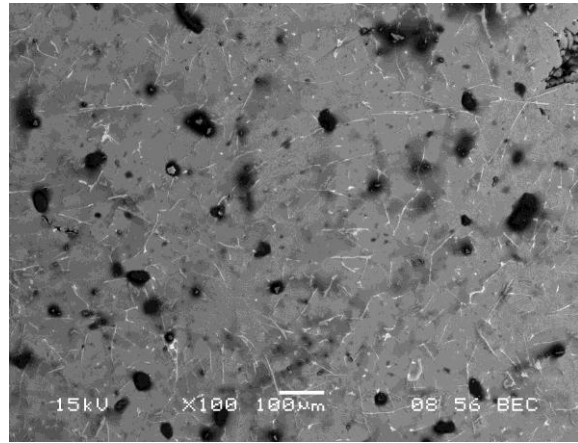


Fig.4.14- Al+2%Mg+10% fly ash composite

The SEM micrographs of Al-fly ash composites are shown in fig 4.8 to 4.14.

The size, density, type of reinforcing particles, and its distribution have a pronounced effect on the properties of particulate composites. The variables affecting the distribution of particles are solidification rate, fluidity, type of reinforcement, and the method of incorporation. It is essential to get particles uniformly throughout the casting during particulate composite production. The first task is to get a uniform distribution of particles in the liquid melt and then to prevent segregation/agglomeration of particles during pouring and progress of solidification. One of the major requirements for uniform distribution of particles in the melt is its wettability.

4.4. Distribution of particles in MMCs

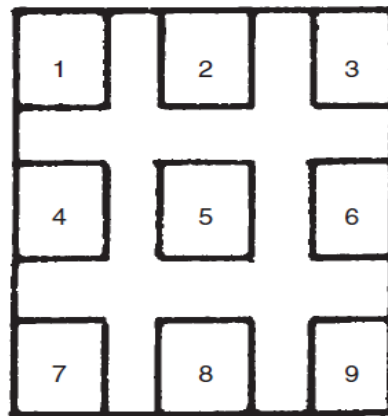
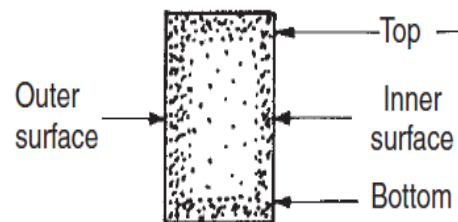
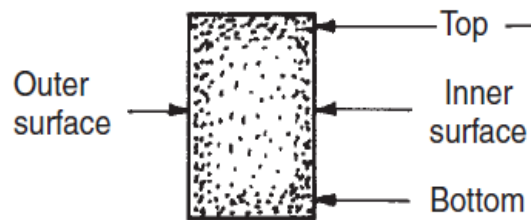


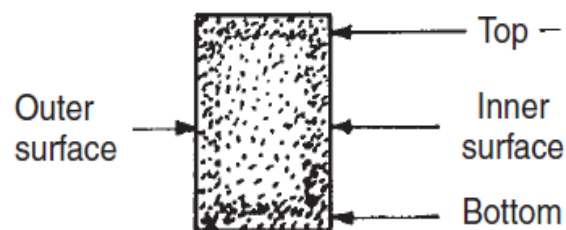
Fig 4.15(a) Different locations of selected sample



Al-10% Fly ash composite



Al+2% Si+10% Fly ash



Al+2% Mg+10% Fly ash

Fig.4.15 (b) Macrographs showing distribution of particles

The micro structure of the samples, cut from the plate casting at different locations, were observed to study the particle distribution (fig 4.15.a). In the case of Al–10% Fly ash, particles were not uniformly distributed throughout the casting. The particles were segregated at the top, bottom, and sides of the plates. The interior of the casting contained very few particles, whereas in the case of Al–2% Mg–10% fly ash and Al–2% Si–10% fly ash particles were present more or less throughout the casting. The particle distribution strongly influences the density of composites. Thus, the density distribution can be used as a measure of particle distribution. In case of Al–10% fly ash, particle rejection was also high. Figure 4.15.b shows schematically the particle distribution in the cast plates.

4.5. Mechanical properties of cast composites

Table 4.2 Hardness

Sl No.	Samples Designation	Hardness (BHN)
1	As cast Al	58.31
2	Al+10%fly ash	67.05
3	Al+2%Si+10%fly ash	70.09
4	Al+2%Mg+10%fly ash	85.95

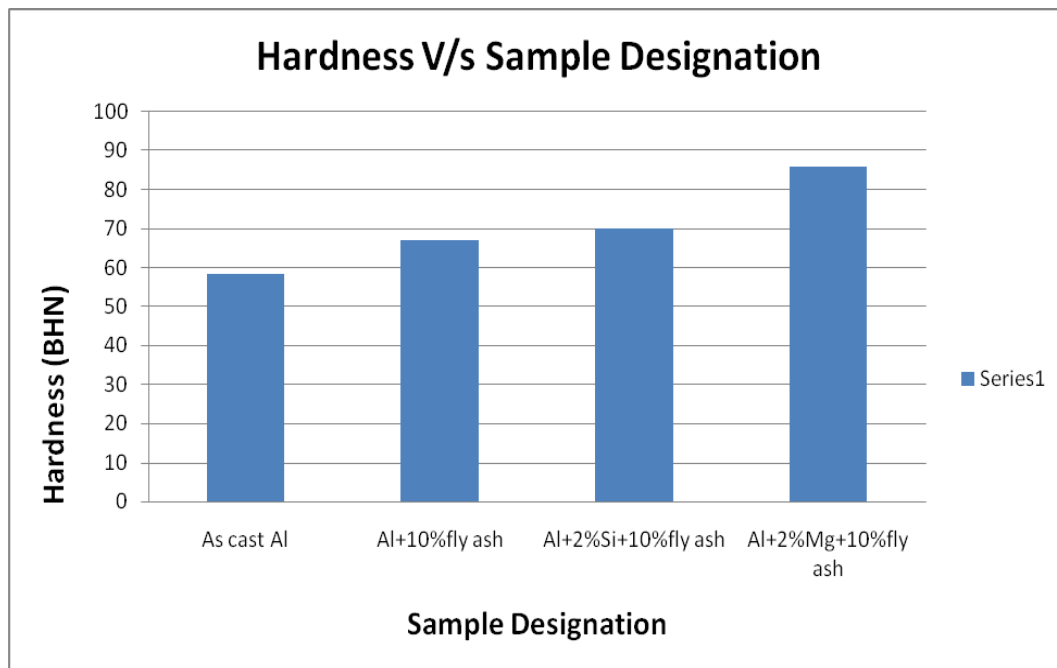


Fig.4.16- Graph showing variation in hardness with composition of MMCs

The above table shows that incorporation of fly ash particles in Aluminum matrix causes reasonable increase in hardness. The strengthening of the composite can be due to dispersion strengthening as well as due to particle reinforcement. Thus, fly ash as filler in Al casting reduces cost, decreases density and increase hardness which are needed in various industries like automotive etc.

Table 4.3.Tensile properties composites

Sl No.	Samples Designation	Hardness (BHN)	U.T.S (Mpa)	% Elongation
2	Al+10%fly ash	67.05	129	4
3	Al+2%Si+10%fly ash	70.09	145	3
4	Al+2%Mg+10%fly ash	85.95	159	2

This indicates that the fly ash addition leads to improvement in the ultimate tensile strength. Form the table it is clear that addition of Mg improve the tensile properties of the composite.

The size range of the particles is very wide. The size ranges of the fly ash particles indicate that the composite prepared can be considered as dispersion strengthened as well as particle reinforced composite. As is seen from the particle size distribution there are very fine particles as well as coarse ones (1-100 μm). Thus the strengthening of composite can be due to dispersion strengthening as well as due to particle reinforcement. Dispersion strengthening is due to the incorporation of very fine particles, which help to restrict the movement of dislocations, whereas in particle strengthening, load sharing is the mechanism.

4.6. Wear behavior

Wear behavior of different composite was studied with different parameter like sliding velocity and applied loads. There result and discussion are given in the following subsections.

4.6.1. Effect of sliding distance

For the entire composites the sliding velocity is 1m/sec and the total running time is 30min with a constant load of 10N.

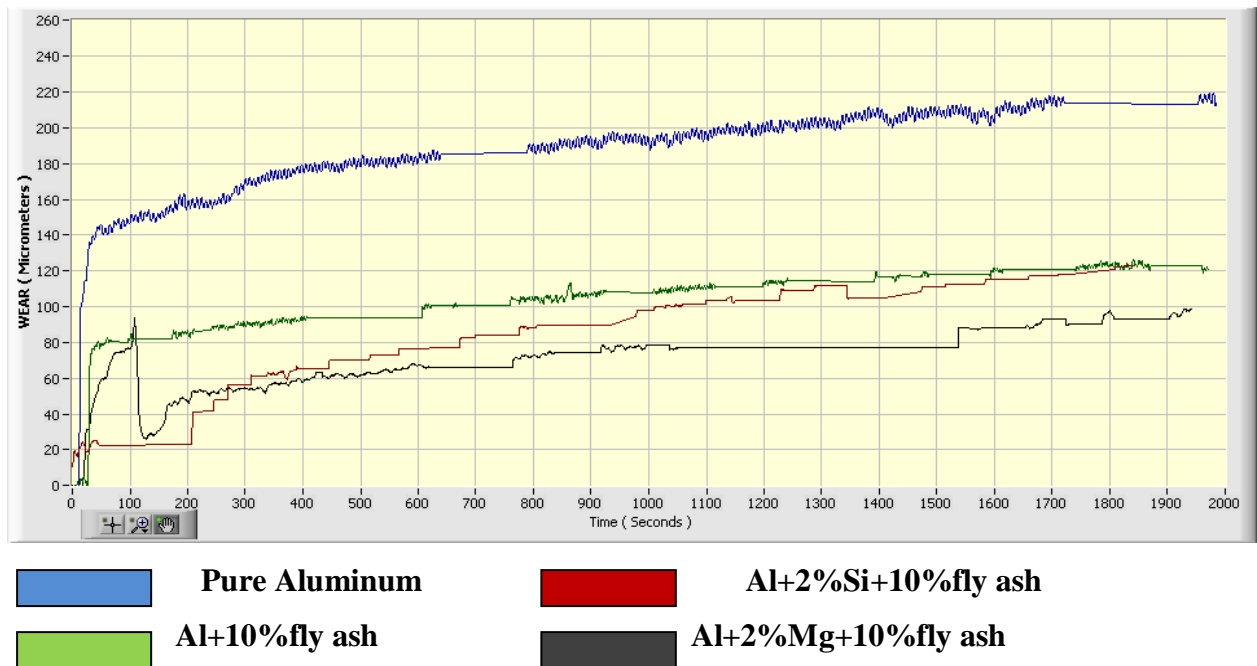


Fig.4.17-Wear behavior of MMCs with different composition under normal load of 10N

Figs. 4.17, 4.18 and 4.19 show of wear curves of MMCs specimen with 10% fly ash, 2% 10% fly ash with 2% Si and 10% fly ash with 2% Mg content with normal load of 10N, 15N, 20N. All the MMCs showed a very small initial nonlinear wear regime. After a certain sliding distance, the wear has increased linearly with time indicating steady state wear regime. The transition from initial wear regime to steady state regime has taken place within a few minutes (2–3 min) of commencement of the test. From the graph (Fig.4.17) it is evident that the wear resistance of composite is much greater than the commercially pure aluminium. Bulk wear decreases with addition of magnesium. Incorporation of fly ash content significantly reduces wear. This evidence from the amount of wear observed for commercially pure aluminum and composite with 2% Mg + 10% fly ash content. This is because of the presence of hard fly ash particles which will increase overall bulk hardness.

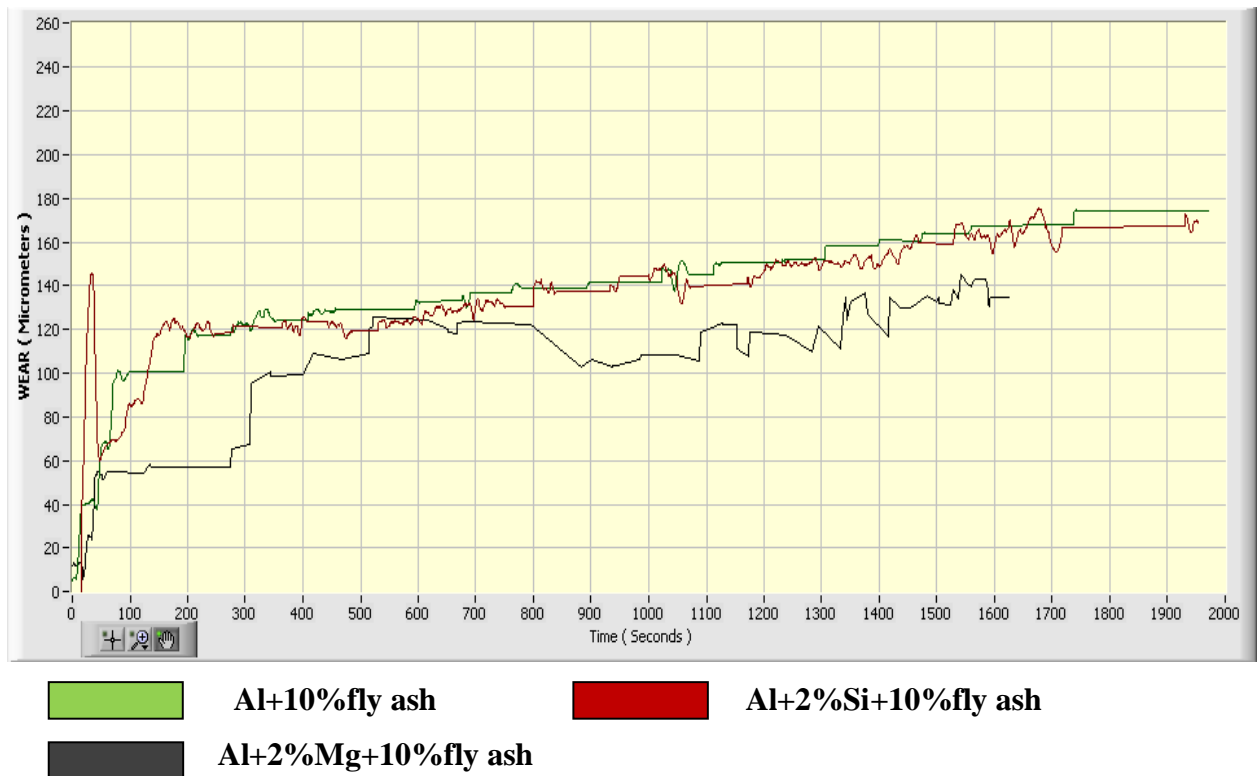
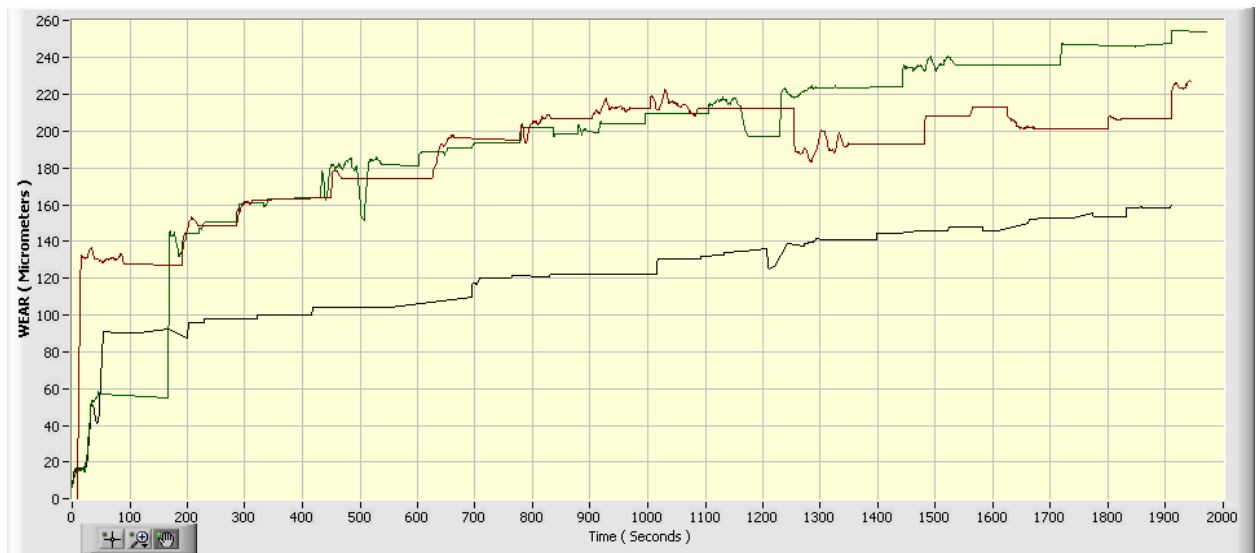


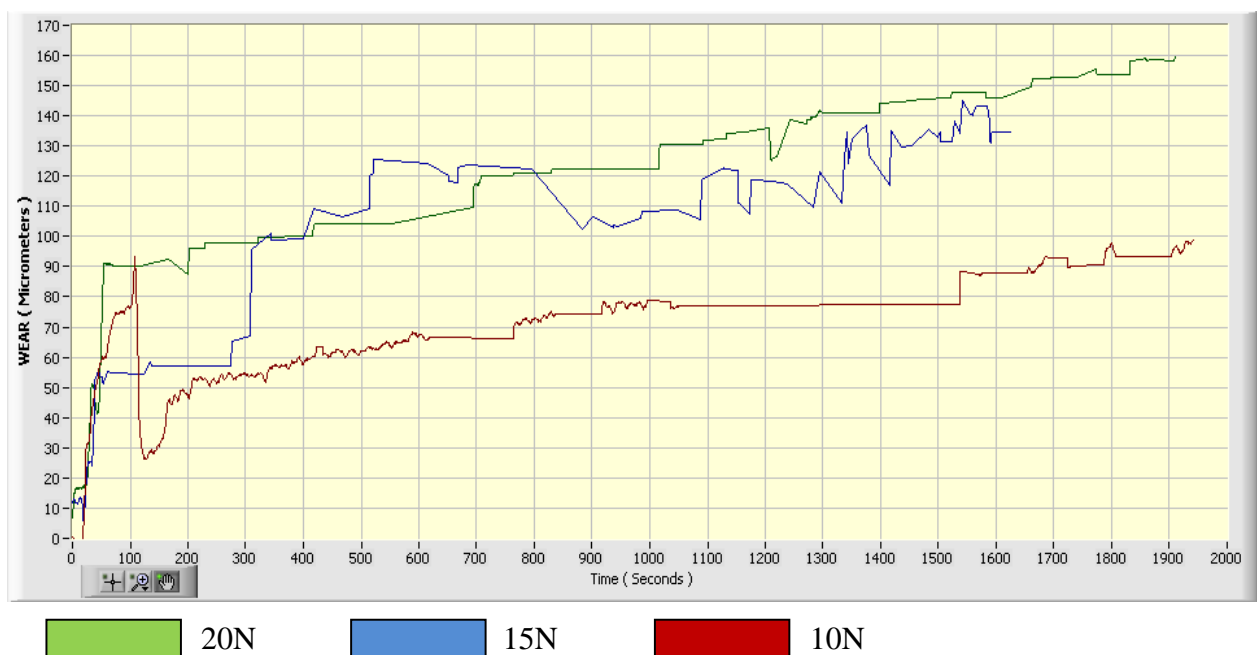
Fig.4.18-.Wear behavior of MMCs with different composition under normal load of 15N

In the initial wear regime, the reinforced particles act as load carrying elements and as inhibitors against plastic deformation and adhesion of matrix material. In the later stages of wear regime, the worn particles get dislodged from their positions in the matrix and get mixed with the wear debris. The wear debris containing matrix material, worn particles and Fe from the disc get pushed into the craters formed by dislodging of particles and act as load bearing elements.



Al+10%fly ash
 Al+2%Si+10%fly ash
 Al+2%Mg+10%fly ash

Fig.4.19-Wear behavior of MMCs with different composition under normal load of 20N



20N
 15N
 10N

Fig.4.20- Wear behavior of MMCs with variation in normal load

Fig.4.20. Shows the relation between applied load and wear of the MMCs (Al+2%Mg+10%fly ash) produced for a particular velocity 1m/sec. The amount of wear has been increased with increase in normal load.

4.6.2. Effect of variation sliding velocity

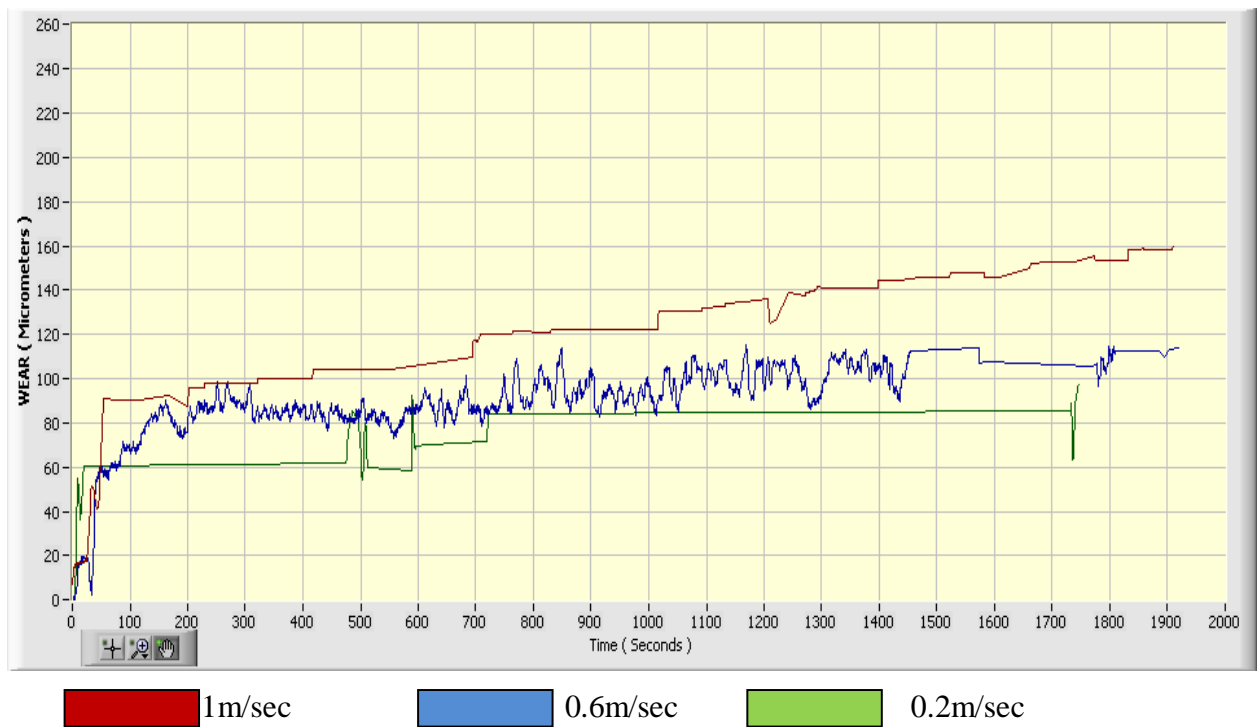


Fig.4.21- Variation of behavior of MMCs with variation in sliding velocity

Variation in sliding velocity was achieved by varying rotational speed of the disc with different rpm keeping track diameter constant. The wear of the composite is significantly increase with increase in sliding velocity.

4.7. Frictional characteristics

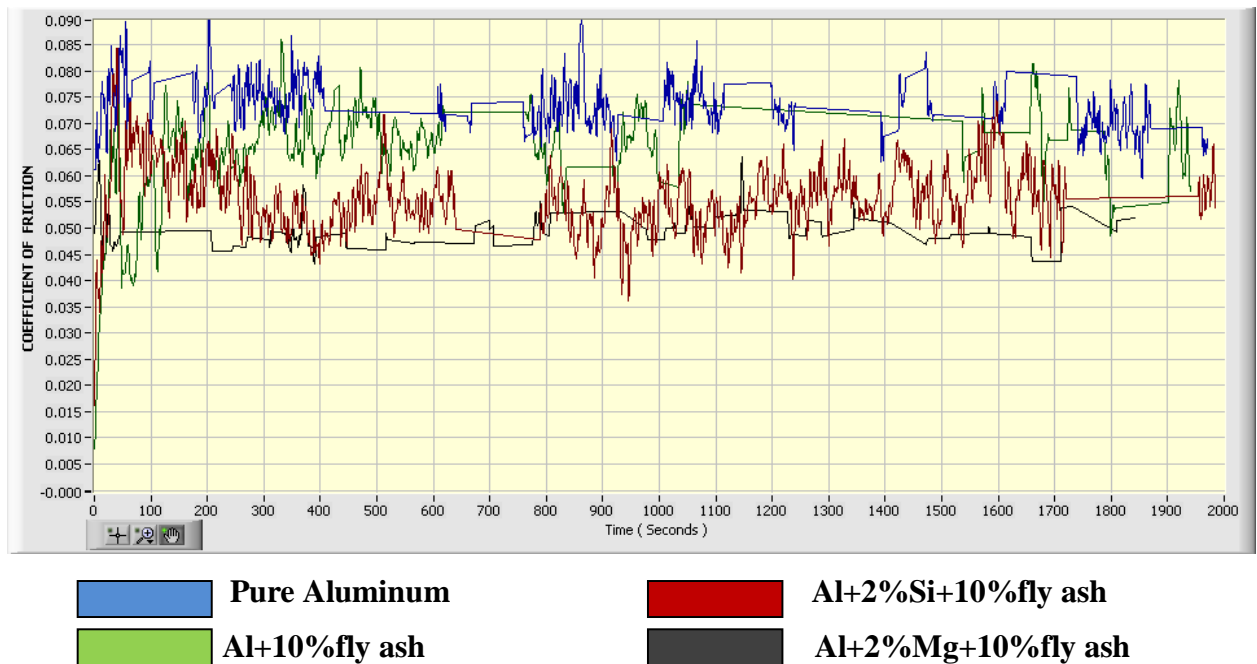


Fig.4.22- Variation of coefficient of friction with composition of the composite

The coefficient of friction has been decreased with increased fly ash content as evident from Fig.4.22. In the test of normal load of 10N and sliding velocity of 1m/sec were used. Composite (Al+2%Mg+10%fly ash) showed lowest coefficient of friction 0.05 and commercially pure aluminium exhibited a coefficient of friction range 0.07 to 0.09. The presence of cenosphere particles will reduce the friction by providing point contact counter face and pin. The coefficient of friction may have reduced with the increase in cenosphere particles in fly ash as the presence of cenosphere particles will reduce the friction by providing point contact between counter face and pin.

4.8. Microscopic examination

Optical microscopic examination of the worn pin surfaces identified different wear mechanism operating under various sliding conditions. In the following sections the observed wear mechanisms are discussed in relation to the sliding conditions and wear rates for better understanding of the tribological characteristics.

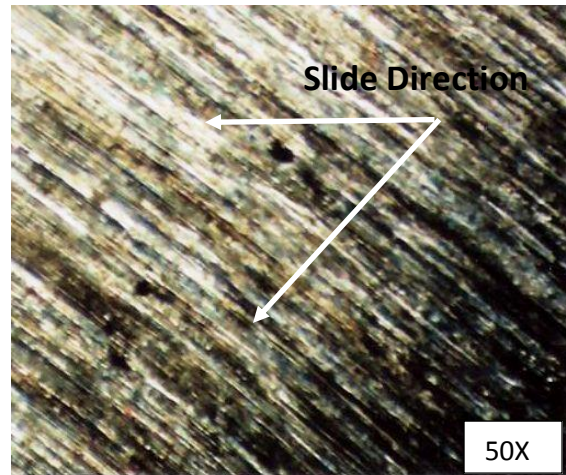


Fig.4.23-Grooves and scratch marks indicating abrasion wear

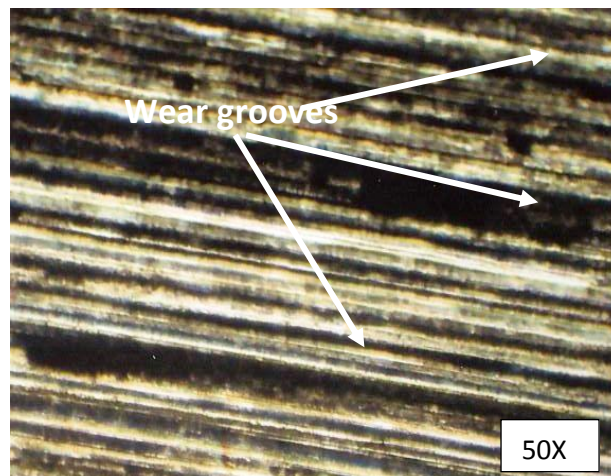


Fig.4.24- Deep smooth and large grooves under higher loads

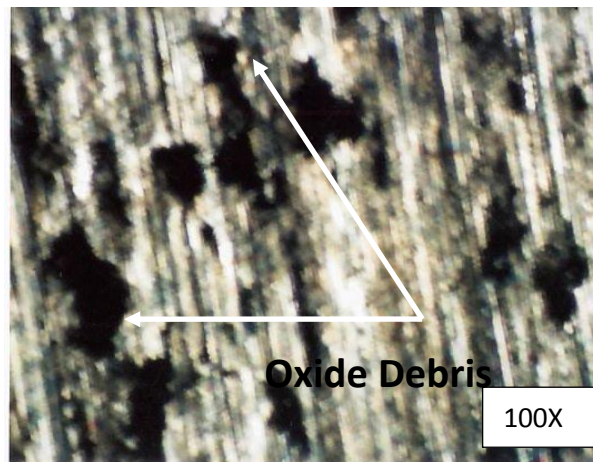


Fig.4.25- Wear debris filling the valleys in the wear surface

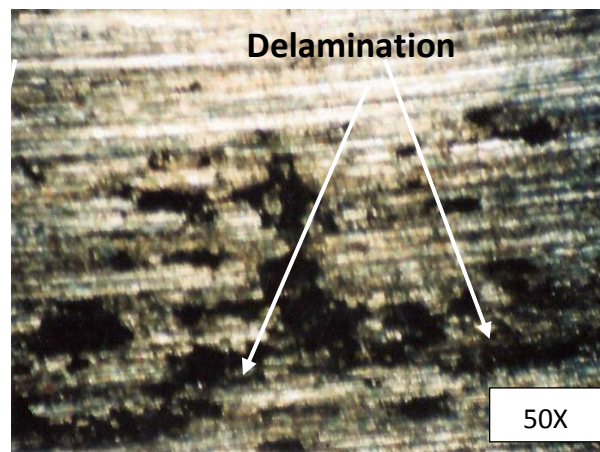


Fig.4.26- Delamination of wear surface under a load of 20N

Abrasion

Most of the grooves are parallel to the sliding direction; it is evident from the worn pins (fig.4.23). Grooving and scratching appear more severe at the higher loads of 20N and higher sliding velocity of 1m/sec (Fig. 4.24). Grooves were less severe for lower loads. Such features are characteristics of abrasion, in which hard asperities of the steel counter face, or hard reinforced particles in between the contacting surfaces, plough or cut into the pin, causing wear by the removal of small fragments of material. The abrasion took place primarily via ploughing, in which material is displaced on either side of the abrasion groove without being removed. The abrasion is extensive in Al/fly ash composites tested, due to the presence of dislodged and fractured fly ash, that become trapped in the sliding interface gets embedded in the counter face, contributing to abrasive wear. In addition, fractured fly ash particles trapped between the sliding surfaces would also cause abrasion of the steel disc, as would work-hardened fragments of matrix alloy and steel.

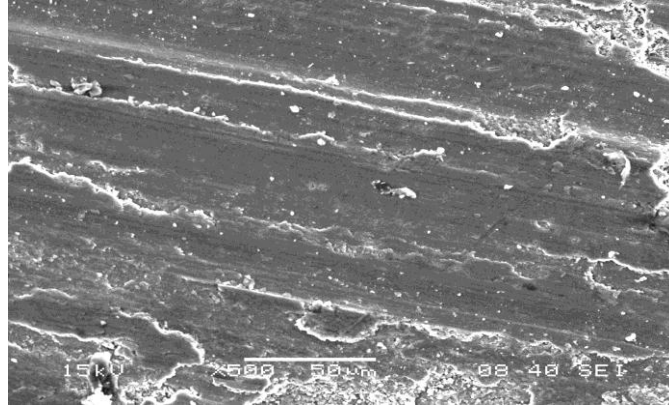
Oxidation

From optical microscope observation, the dark surfaces are found to be covered extensively by a thin layer of fine particles. Large amounts of fine powder are also present in the wear debris. These characteristics are indicative of oxidative wear, in which frictional heating during sliding causes oxidation of the surface, with wear occurring through the removal of oxide fragments. Over continuous sliding, oxide wear debris fills out the valleys on the pin surface, and gets compacted into a protective layer preventing metallic contact and wear rate of the composite drops accordingly Fig. 4.25.

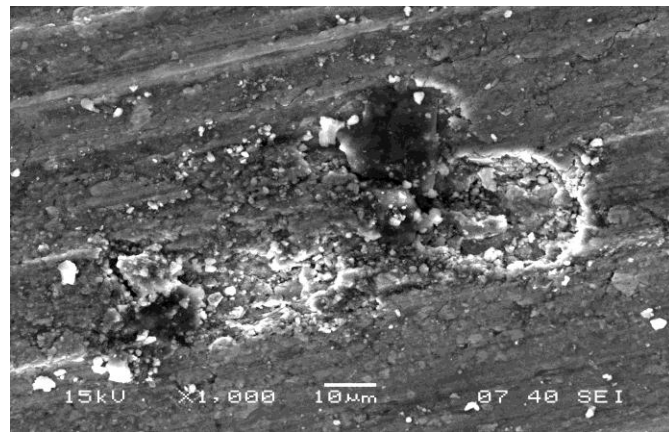
Delamination

For higher loads of 20N with speed of 1m/sec and the MMCs (Al+2%Mg+10%fly ash), the wear mechanism has been linked to the process of delamination in fig 4.26. This is a fatigue-related wear mechanism in which repeated sliding induces subsurface cracks that gradually grow and eventually shear to the surface, forming long thin wear sheets. Delamination is observed to be more extensive under the higher load of 20N.

4.9. SEM micrographs of the worn surface the composite



(a)

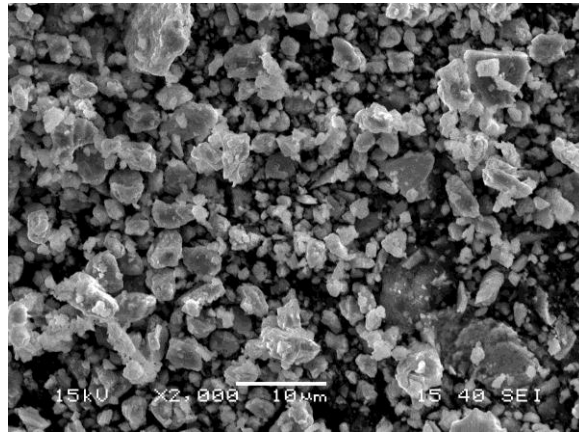


(b)

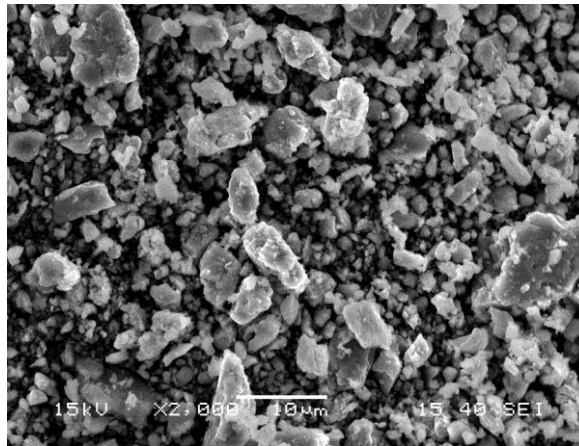
Fig.4.27—SEM micrographs of the worn surfaces after the wear test under velocity 1 m/s; time 30min; (a) load 10N and (b) 20N.

Examination of the worn surfaces of aluminum–fly ash composites under the SEM after wear test shows that under the load of 10N, the worn surface has relatively less ploughing and cutting, as shown in Figure 4.27(a). However, at the load of 20N, fractured fly ash particles are frequently present on the worn surface, as shown in Figure 4.27 (b).

4.10. Wear debris



(a)



(b)

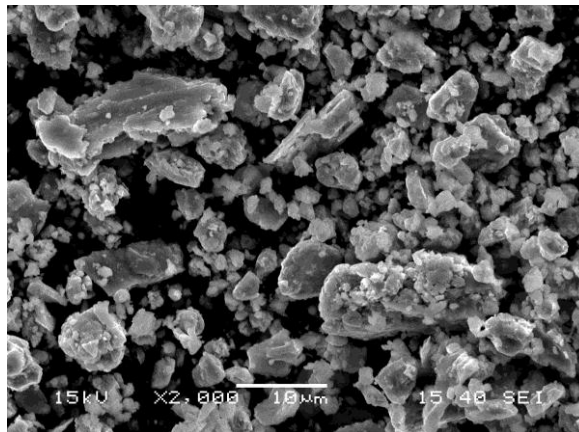
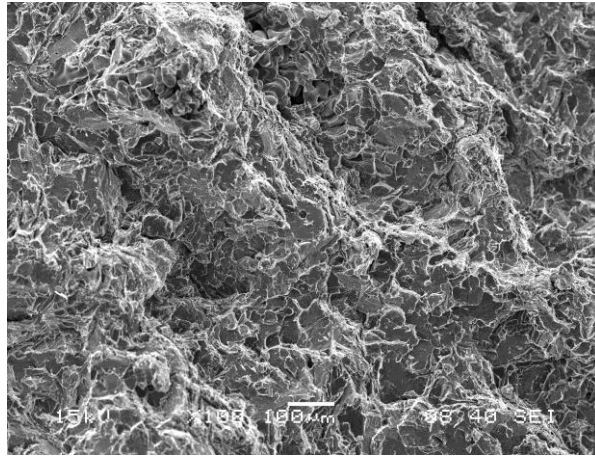


Fig.4.28-SEM micrographs of debris of the MMCs at different loads at (a) 10N, (b) 15N and (c) 20N

The material is transferred back and forth several times during the sliding process and eventually produces particles of wear debris. When the applied load results in stresses higher than the fracture stress of fly ash particles, these particles lose their ability to support the load. Consequently, the aluminium matrix comes in direct contact with the counter face and large plastic strains are imposed on the contact surfaces of the pin. The severe localized deformation gives rise to crack formation, a process in which fly ash particle/matrix decohesion plays an important role. Surface delamination is also contributed to process of debris formation. Visual observations indicate wear debris are dark in colour.

A substantial quantity of wear debris was generated during the tests. Two major morphologies, particle-like or flake- observed in all tests, the only difference is the relative amount of each type, and this could be influenced to a great degree by the normal load and sliding velocity. The particle size and irregularity increased with the applied load. The fig 4.27 shows that the size of the particles larger and irregular for 20N applied load as compared with 15N and 10N load. The volume of the wear debris increases with increasing normal loads and sliding velocity, thereby resulting in greater wear loss for higher loads and sliding velocities.

4.11. Fractograph of the selected composites



(a)

Fig4.29-SEM fractograph of Al+2%Mg+10% fly ash

The tensile fracture surface of fly ash reinforced is shown in fig 4.29. The fractograph taken in SEM revealed absence of dimples which are indicative of ductile fracture. This observation is consistent with mechanical properties results which show a reduction in elongation. (Table no 4.3).

CONCLUSIONS

The following conclusion may be drawn from the present work:

- From the study it is concluded that we can use fly ash for the production of composites and can turn industrial waste into industrial wealth. This can also solve the problem of storage and disposal of fly ash.
- Fly ash up-to 20% by weight can be successfully added to commercially pure aluminium by stir casting route to produce composites.
- Addition of magnesium and silicon improves the wettability of fly ash with aluminium melt and thus increases the retention of the fly ash in the composite.
- Hardness of commercially pure aluminium is increased from 58BHN to 86BHN with addition of fly ash and magnesium.
- The Ultimate tensile strength has improved with increase in fly ash content. Whereas ductility has decreased with increase in fly ash content.
- The effect of increased reinforcement on the wear behavior of the MMCs is to increase the wear resistance and reduce the coefficient of friction. The MMCs exhibited better wear resistance due to its superior load bearing capacity.
- The wear resistance of composites is much greater than the commercially pure aluminium.
- Different wear mechanisms were found to operate under the test conditions of variation of normal loads, composition, and sliding velocity. They are oxidation abrasion and delamination.
- Increased normal load and sliding velocity increases magnitude of wear and frictional force.

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